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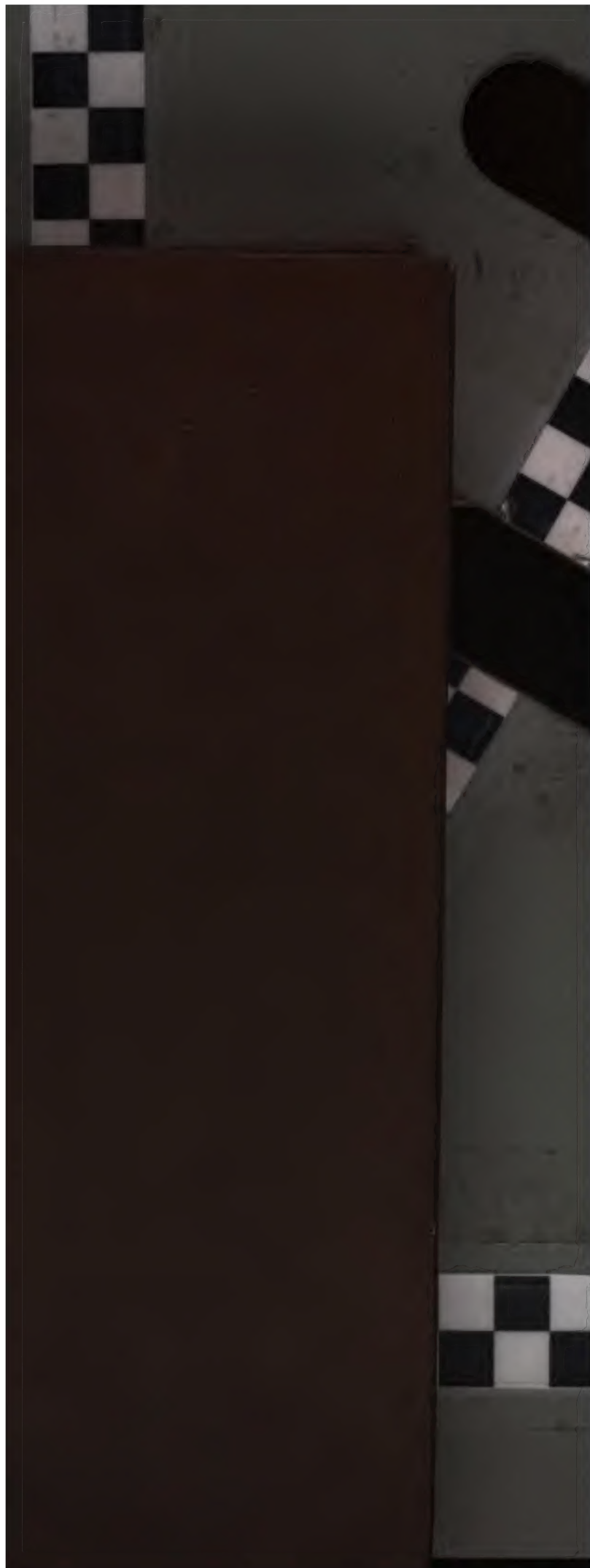
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# The Analyst,

INCLUDING THE PROCEEDINGS OF

THE "SOCIETY OF PUBLIC ANALYSTS."

A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT OF THE  
ANALYSIS OF FOOD AND DRUGS, AND OF GENERAL  
ANALYTICAL AND MICROSCOPICAL RESEARCH.

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EDITED BY

JOHN MUTER, M.A., Ph.D., F.R.S.E., F.I.C., F.C.S., ETC.,

*Late President of the Society of Public Analysts ; Author of "A Short Manual of Analytical Chemistry,"  
and of Text-Books on Pharmaceutical Chemistry and Materia Medica ; Public Analyst for  
Lambeth, Wandsworth, Southwark, Bermondsey, Rotherhithe, Newington,  
Tenterden, etc., Analyst to the Metropolitan  
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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of the Society was held at Burlington House on Wednesday, the 8th ult., the President, Dr. A. Hill, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened it was announced that the following gentlemen had been elected as members:—J. M. H. Munro, D.Sc., F.C.S., of Downton Agricultural College; A. G. Salamon, A.R.S.M., F.C.S., London; Dr. John A. Voelcker, B.A., B.Sc., chemist to the Royal Agricultural Society.

The following gentlemen were proposed for election, and will be balloted for at the annual meeting:—As member, H. Faber, chemist to the Dairy Supply Company; as associate, Adolphus Dretel, assistant to Mr. W. Johnstone.

Mr. Harland and Mr. Johnstone were appointed auditors, to examine the accounts for the past year.

The following papers were read and discussed:—

Notes on the Alcoholic Fermentation of Milk-Sugar, by Dr. P. Vieth, F.C.S.

A new Method of ascertaining the amount of Fat in Milk, by H. Faber.

Experiments with the Lactocrite, by A. Wynter-Blyth, M.R.C.S., F.C.S.

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On Reichert's Butter Method: a Correction, by A. H. Allen, F.C.S., F.I.C.

Notes on Thickened or Blown Oils, by J. Baynes, F.C.S., F.I.C., and W. Fox, F.C.S.

The annual meeting of the Society will be held at Burlington House, on Wednesday, January 12th, and the annual dinner will take place the same evening. The usual notice will be given to members. Mr. Young's paper on the Logwood Test, that of Messrs. Baynes and Fox on Blown Oils, and Mr. Wynter Blyth's on the Lactocrite, will all appear in our next issue.



## NOTES ON THE ALCOHOLIC FERMENTATION OF MILK-SUGAR.

By P. VIETH, PH.D., F.C.S.

*Read at the Meeting, December 8th., 1886.*

A PAPER ON "The Composition of Mares' Milk and Koumiss," which I read before this Society at the November meeting, 1885, gave rise to a conversation on the alcoholic fermentation of milk-sugar, in the course of which it was remarked that there exists some difficulty to start alcoholic fermentation in solutions of milk-sugar.

In order to convince myself of this fact, I made the following simple experiment: 5 per cent. solutions of cane-sugar and of milk-sugar were made, and German yeast added to each of them, in the proportion of 1 gramme to 100 c.c. The solutions were kept loosely covered side by side in a moderately warm place, the temperature of which varied from 16° to 28°C. Samples—5 c.c.—were taken daily, evaporated on the steam bath, dried in the air bath at 100°C., and weighed. The cane-sugar solutions were on the evening of the first day already in active fermentation, and the solids diminished rapidly during the early part of the experiment, viz., about at the rate of 30 per cent. per day, the residue of 5 c.c. of the solution weighing, when freshly made, .2574, after one day .1674, after two days .1150, after three days .0787. In the solution of milk-sugar, alcoholic fermentation was not perceptible; there was, however, also a slight diminution of solids. The solutions contained solids:—

	I. Series.		II. Series.	
	Cane-Sugar.	Milk-Sugar.	Cane-Sugar.	Milk-Sugar.
When fresh	.. 5.14	4.96	5.36	5.16
4 days old	.. 1.18	4.90	1.38	5.00
8    "   "	.. .62	4.80	.64	4.96
12   "   "	.. .52	4.66	.58	4.90

From these figures it appears that within twelve days the solids of the cane-sugar solution were reduced to about one-tenth of the original amount, while those of the milk-sugar solution diminished by about 6 per cent. only.

I have never tried the experiment to keep a plain solution of milk-sugar, without or with yeast, added to it, for a longer period, and see if alcoholic fermentation would have set in, but done so with a preparation of milk containing as much milk-sugar and as little of the other solid constituents as possible—whey. Milk set in shallow pans will turn sour and thick in two or three days, if kept at mean temperature. If the milk is then slowly heated the precipitated casein is contracted and forms lumps of tough curd, while whey is separated, which, provided poor skim milk had been used, will be almost clear. Whey prepared in this manner in November, 1885, was bottled immediately after having separated and cooled down, the bottles corked and wired and removed to a cool vault. The whey was analysed when it was six and twelve months old; after six months already it was found to be highly effervescent. The whey contained:—

	6 Months Old.	12 Months Old.
Total Solids	.. 6.28	5.46
Lactic Acid	.. 1.73	2.58
Alcohol	.. .63	.93

In May last I repeated the experiment, and at the same time put aside some whey



pared of sweet milk by means of rennet. The two kinds of whey were analysed when fresh and six months old. The sour whey again was found highly effervescent when six months old, but the whey prepared by means of rennet contained a small quantity of gas only. The results of analysis were as follows :—

	Sour Whey.		Sweet Whey.	
	Fresh.	6 Months Old.	Fresh.	6 Months Old.
Total Solids	.. 7.51	5.88	6.84	6.24
Lactic Acid	.. .65	2.38	—	1.47
Alcohol	.. —	.53	—	.20

These experiments show that spontaneous alcoholic fermentation sets in if whey is kept for a considerable length of time, and, in the case of sour whey, develops to a very marked degree.

I expected that the fermented whey would act as a powerful ferment on milk-sugar solution, and in order to try it made two 5 per cent. solutions of milk-sugar containing twelve months old fermented whey, the one in the proportion of ten and the other of twenty per cent. The solutions were kept loosely covered at a temperature varying from 16° to 28°C., and the solids determined daily. Instead of a decrease, I, during the early part of the experiment, found an increase in solids, due, no doubt, to the evaporation of alcohol introduced with the fermented whey; after a time the solids very slightly diminished. The percentages of solids found were as follows :—

	5 per cent. solution of milk-sugar +	
	10 per cent. old whey.	20 per cent. old whey.
Newly prepared	.. 5.06	5.40
After 4 days	.. 5.08	5.54
„ 8 „	.. 5.12	5.22
„ 11 „	.. 5.02	5.06

In my paper “On Mares’ Milk and Koumiss,” I have mentioned that mares’ milk spontaneously undergoes not only lactic but also alcoholic fermentation, sometimes within a very short time; I have seen mares’ milk, milked at 8 a.m., frothing with carbonic acid gas at 6 p.m. of the same day. In the manufacture of koumiss alcoholic fermentation is supported or effected by the addition of koumiss, which is well fermented. It is well known that a beverage, similar to and having all the properties of mares’ milk koumiss, can be prepared of cows’ milk. During the last few years attention has frequently been drawn to a new preparation, consisting of fermented cows’ milk, and known under the name of “Kefir.” In this case alcoholic fermentation is started in the milk by the addition of a ferment called kefir grains. These grains contain, according to Dr. Kern, of Moskow, a bacillus, to which he gave the name of *Diospora Caucasica*, and yeast cells, apparently a modified form of *Saccharomyces Cerevisiae*. Milk, to which kefir grains have been added, and which is subjected to a certain treatment, undergoes alcoholic as well as lactic fermentation within an exceedingly short time. After one day the alcohol found in kefir amounted to .3, after two to three days to .8, after five days to 1 per cent.

I dare say every Public Analyst has made the experience that milk samples kept for some length of time get fermented, developing gaseous products. That the solids in such samples decrease to a considerable degree has frequently been discussed. Some

years ago I brought before the Society\* some observations relating to the speedy diminution of solids in milk, which is kept in small quantities at mean temperature, and allowing the free access of air to it. I then suggested that this diminution in solids must be attributed to alcoholic fermentation, and am of the same opinion still. One might think of a decrease caused by the formation of di-lactic acid on drying up sour milk, but supposing the whole of the milk-sugar be converted in lactic acid—which is highly improbable—and the whole of the lactic acid in di-lactic acid, which is impossible under the circumstances—then a loss could be caused only equal to .45 to .50 per cent. in milk containing 4.5 to 5.0 per cent. milk-sugar.

I have repeated the experiment of keeping small quantities of milk in platinum dishes exposed to the air in a moderately warm place—about 25° C.—for varying periods of time, and compared them as to the amount of solids with other samples of the same milk kept in a stoppered bottle, but otherwise under the same conditions. At the same time I tried to find out what effect the addition of yeast—1 gramme to 100 c.c.—would have upon the milk under those conditions. The experiment, for certain reasons, was made with very poor sweet skim-milk. The following results were obtained:—

Solids in skim-milk kept in stoppered bottles.				
		Without yeast.	With yeast.	
Fresh ...	...	9.80	10.08	(Lactic Acid .15)
After 4 days...		9.48	9.70	( " 1.01)
" 8 "	...	9.32	9.53	( " 1.16)
" 11 "	...	9.32	9.31	( " 1.21)
Solids in skim-milk kept in platinum dishes.				
		Without yeast.	With yeast.	
4 days	...	8.80		9.04
8 "	...	8.34		8.40
11 "	...	8.30		8.10

With regard to the samples taken from the bottles, it must be remarked that particles of precipitated casein stuck to the glass, and could not be removed, not even by violent shaking, a circumstance which accounts for the relatively great difference between the milk when fresh and four days old. Otherwise, the results want no commentary.

On reviewing the facts which have been brought forward we see, on the one hand, the difficulty of starting alcoholic fermentation in pure solutions of milk-sugar as well as in milk by means of added yeast; on the other hand, alcoholic fermentation readily developed in milk by a certain ferment; further, alcoholic fermentation setting in spontaneously in whey and milk, if kept for some considerable length of time; and a very marked diminution of solids within a very short time, which seems to be attributable to alcoholic fermentation, if milk is kept in small quantities exposed to the air in a moderately warm place.

I do not think I need apologise for bringing these observations under your notice, as they have a direct bearing on the much-discussed question of analysing old samples of milk, but I have to apologise for leaving the question at a stage where a more close investigation ought to commence. In common with many analysts engaged in practical work, I do not always find time to further investigate into questions, the clearing up of which is very desirable.

\* THE ANALYST, 1882, p. 213.



## DISCUSSION.

Mr. HEHNER said there was an article in the market something of the nature of a "bland," but it was perfectly transparent, and he understood it was the whey of the milk fermented. It contained no acid, and he should like to ask how it had fermented.

Mr. KINGZETT regretted that he had not heard the early part of Dr. Vieth's communication, but he thought that a brief description of some results which he had obtained with reference to the fermentation of milk and milk-sugar some time ago might have some general interest for the members. The experiments in question formed part of an exhaustive investigation, made with a special object, and were as follows:—

A quantity of fresh milk was taken, and the milk-sugar present in it was determined by first of all diluting 10 c.c. with 90 c.c. of water, and then estimating it by the well-known process which makes use of Fehling's solution volumetrically. It contained 4.422 grammes milk-sugar in each 100 c.c. It was also experimentally proved to be quite free from lactic acid. 50 c.c. of this milk was diluted with 50 c.c. of water, and the mixture placed in a flask loosely covered with filter paper, on July 7th. On July 20th, the mixture was gently warmed, to complete coagulation, and was then filtered, the filtrate being made up to a known volume by washing the coagulum with warm water, after which the sugar present in the filtrate, and the acidity thereof, were respectively determined.

Sugar originally present.	Sugar present at end.	Difference due to fermentation.	Final Acidity.
2.211 grammes.	None.	2.211 grammes.	28 c.c. $\frac{N}{10}$ NaHO.

A microscopic examination of the fermented mixture revealed the presence of the *bacterium lactis* in great numbers, and the *bacterium termo* was also seen to be present in a very active state. As the fermentation of 2.211 grammes of milk-sugar should yield, if entirely fermented into lactic acid, the same weight of that substance, whereas the acidity of the fermented mixture was only equal to 0.252 gramme of that substance, it was concluded that the lactic acid had itself been resolved by subsequent changes into acetic, butyric and propionic acids, and that, finally, these acids had been split up by oxidation into carbonic anhydride and water; moreover, as the fermentation was conducted fortuitously, it doubtless was not of a pure character.

*Experiment 2.* This was made with another sample of milk, containing 3.88 grammes milk-sugar in each 100 c.c.; the mixture being allowed to stand from August 4th to August 13th.

Sugar originally present.	Sugar present at end.	Difference due to fermentation.	Final acidity.
1.940	0.980	.960	37.5 c.c. $\frac{N}{10}$ NaHO

In this experiment the quantity of lactic acid found is greater in proportion to the sugar employed, and, doubtless, this result was due to the fact that the milk was not allowed to stand so long as in the previous experiment. If the whole of the milk-sugar which had disappeared had been changed into lactic acid, and none of this had been destroyed by subsequent chemical changes, there would have been obtained 0.96 gramme lactic acid, instead of 0.3375, actually calculated to be present.

*Experiment 3.* This experiment was conducted as follows:—Some 245 c.c. of fresh milk was inoculated with 5 c.c. of very sour milk; then the acidity and the milk-sugar of the inoculated mixture were carefully determined; after which two quantities of milk 10 c.c. each were respectively diluted with 50 c.c. water, and allowed to stand from August 20th to August 24th.

Sugar originally present.	Sugar present at end.	Difference due to fermentation.	Original acidity.	Acidity at end.
A. 2.185	1.675	.510	0.5 c.c.	44.5 c.c. $\frac{N}{10}$ NaHO
B. 2.185	1.654	.530	0.5 c.c.	45.7 c.c. $\frac{N}{10}$ NaHO

The results of the examination of the two mixtures are seen to be thoroughly and mutually confirmatory in character. Taking A, the loss of the milk-sugar could produce a maximum of .510 gramme lactic acid, whereas the acidity of the ultimate mixture was equal to .4005 gramme, leaving apparently only .1095 gramme to be accounted for. It is, however, to be noted that the approximation of the theoretical and practical quantities of lactic acid is much greater than in either of the previous experiments, and doubtless the reason is that the mixtures were only allowed to stand over four days. In order to test the supposition that alcohol might also be found amongst the products, thus accounting for the difference, one half of each mixture A and B was subjected to distillation, and the specific gravity of each distillate after being made up to the original volume, was determined and found to be respectively .99977 and .99973. Alcohol was thus proved to be present in each fermented mixture, in more than sufficient amount to fully account for the deficiency in lactic acid produced. The explanation is this: the alcohol which was found present was all derived from the fermentation of lactose; whereas a small proportion of the total acidity of mixtures A and B was derived from butyric and other soluble fatty acids, which resulted from the rancidity of the cream or fat contained in the milk, thus swelling the amount of acid which is, as explained, not derived solely from the lactose.

Either, then, the *bacterium lactis* performs the chemical changes whereby lactose is resolved both into alcohol and lactic acid, or it was accompanied in these experiments with other forms of microscopic life, which fermented a part of the lactose into alcohol and carbonic anhydride.

Dr. VIERI, in reply to Mr. Hohner, said there was no impossibility about having a preparation which might contain whey, and might ferment at the same time, but he did not think it was suggested that the preparation referred to consisted entirely of whey.

What had principally struck him was, on the one hand, the great difficulty of starting fermentation of milk-sugar by adding yeast as the fermenting power, and, on the other hand, the quick fermentation by adding some other ferment.

## A NEW METHOD OF ASCERTAINING THE AMOUNT OF FAT IN MILK.

By H. FABER.

*Read at the Meeting, Dec. 8th., 1886.*

AN apparatus which would render possible an easy, quick and exact determination of the amount of butterfat in milk, has for many years been a desideratum, but all attempts to construct such an apparatus have till quite recently proved unsuccessful.

The simplest way of estimating the amount of fat, which nature itself seems to suggest, is to let the cream rise and notice its volume. This method, which is extensively used by practical men, is however very unreliable, as there is no constant relation between the volume of cream thrown and the percentage of pure fat. An attempt has been made to improve this method by dissolving the fat in ether, as in the Marchand's Lactobutyrometer, but sufficient accuracy can hardly be obtained in this way. The best apparatus, so far as accuracy is concerned, is undoubtedly Professor Soxhlet's aerometrical apparatus, by which the specific gravity of an ethereal solution of the fat is taken, the solution being made according to a given method and the gravity taken at a fixed temperature. A certain sp. gr. then corresponds to a certain amount of fat in the



milk, but it may reasonably be objected to this method that it is rather costly, and that such a delicate instrument could not well be worked by a practical dairyman.

As far back as 1859, experiments were made by Professor C. J. Fuchs at Carlsruhe with the view of obtaining a more complete and uniform rising of the fat globules by employing centrifugal force instead of the force of gravitation, as in the ordinary creamometer; but he failed to produce a sufficiently strong and rapidly revolving machine. In 1881 Professor N. J. Fjord, of Copenhagen, constructed his "Control Centrifuge," especially intended for, and by him mostly used for examining separated milk, for which purpose it worked to great satisfaction. It was never intended to be used by others besides his staff, for which reason he never published any directions for its use; it was not intended for, nor did he claim that it could be used for analysing whole milk. Nevertheless it has been taken up, exhibited, and at many places used for analysing whole milk. On account of its favourable reception he has lately altered his apparatus, which is now intended for controlling the supply of milk and enabling dairy factories to pay for it according to the amount of butterfat it contains. The apparatus in its new shape contains fifty-four cylindrical tubes, to hold fifty-four samples of milk. The cream is made to rise in the tubes by making the apparatus revolve about 60,000 times in the Danish Centrifuge, which takes about three-quarters of an hour. The thickness of the cream is thus then measured.

There is one objection to be made to this apparatus, viz., that it indicates the amount of cream and not of *butterfat*, and although undoubtedly the completeness of the rising of the cream and its uniformity is much greater in this case than in the ordinary creamometer, still this app. must only be considered as one step further towards the ideal, which is to have the *pure butterfat* isolated by mechanical separation. This is the goal after which Dr. de Laval, of Stockholm, has been striving, and which he has at length attained in his new machine, the Lactocrite.

In order to get the fat globules in the milk to unite to one clear mass of fat, it is necessary to render the casein more completely dissolved than it is in the milk in its natural state. There has been, and probably is still, a difference of opinion as to whether the fat globules are coated with a membrane or not, but all agree upon the fact that for some reason or other they do not unite as readily as might be expected. The truth seems to be that by a molecular attraction the casein forms a condensed layer, but not a real membrane, around the fat globules. To dissolve the casein Dr. de Laval at first tried an admixture of alkali, which proved of little avail. He therefore took the opposite course and succeeded in dissolving the casein completely by boiling the milk with acetic acid. As is well known, small quantities of free acetic acid will precipitate the casein while a large excess will redissolve it. By the proposed treatment the serum of the milk is transformed into a perfectly clear and thin fluid, and the fat is apparently not affected.





The apparatus itself consists of a strong round steel disc on a spindle, like that of the separator bowl, and test-boxes of platina-plated brass with graduated glass tubes. The *modus operandi* is as follows:—10 c.c. of the sample of milk to be tested are run into a small test-glass, afterwards 10 c.c. of glacial acetic acid, containing five per cent. by volume of concentrated sulphuric acid are run into the same glass, which is then closed with a perforated cork-stopper, in which is inserted a piece of glass tube; this serves to prevent a concentration of the contents of the test-glass during the boiling. In a water-bath, arranged to hold twelve test-glasses, these are heated by steam or gas for seven or eight minutes, after which time the casein has been completely dissolved, while the liquid has acquired a slight violet tinge. The next step is to charge the test-boxes. These consist of a cup in which a perforated stopper fits tightly. The stopper holds the graduated glass tube, as the fat in the milk after boiling with acid has a great tendency to rise, the test-glass must be well shaken before its contents are poured into the cup, and when this is filled the stopper must be immediately pressed down in it, whereby any excess of the mixture will escape through the glass tube, and the test box is then filled completely. After the test-boxes have been charged in this way, they are ready to be placed in the disc, which will hold twelve at a time. The disc, which before use must be heated to about 110° F. by being placed in warm water of this temperature, has twelve cylindrical holes bored from a cavity on the top, radiating and a little sloping. In these the test-boxes are placed (if less than twelve test-boxes are used, there should always be an even number placed so as not to disturb the equilibrium) and the cavity is filled with water, which will keep the liquid in the test-boxes from being pressed out by the centrifugal force. The disc, which fits any stand of a Laval separator, is now made to revolve for three or four minutes at ordinary speed (6000 revolutions in the minute). When it is again at rest the test-boxes are drawn out, and the column of fat in the graduated tube is read off, the divisions indicating immediately tenths per cent. of butter-fat by weight.

Before entering into the question of comparative analytical results it will be necessary to say a few words of explanation. It has been stated that any method of determining the amount of fat will give corresponding results in the hands of persons working in the same way and in the same laboratory, and that no method will give the same results on the same sample of milk in the hands of different analysts at different places. The first statement may be right, but is of very little interest; the second would be very serious indeed, if true. Any method which will extract all the fat and nothing else, will give very nearly the same results in the hands of any careful analyst, but it is an essential condition of a good method, that it shall extract *all the fat and nothing else*. All methods possessing these two qualities will give the same results on the same sample of milk carefully worked. To obtain a complete extraction of the fat, the milk must be given a very large surface, but this must not be done on paper containing resinous matter, as something will then be extracted besides the fat. For the same reason the ether used must be redistilled. When using Adams' method it is indispensable that all the resinous matter shall be extracted beforehand, which does not seem very easy. [I have found in one case that 5 siphonings extracted 0.023 gramme of a coil, but still left 0.010 gramme behind, which were extracted by 8 more siphonings.] With well-washed

paper coils I have found that Adams' method will give results corresponding very closely with those obtained by the method I generally use, which was first described by Dr. V. Storch, in 1883, but had then been in use for several years. According to this method about 10 grammes of milk are dried on about 10 grammes of pumice stone, ground to the size of lentils, sifted to remove the dust, and heated to a red heat. The dry mass is finely ground in a porcelain mortar and extracted in a very simple extracting tube. 50 c.c. of redistilled ether may be forced to percolate through the finely pulverized milk any number of times so as to remove all fat, and nothing but the fat can possibly be extracted. In analysing a sample of skim-milk twice by each of these methods I found:—

By Adams' method	...	...	0.70	0.68 per cent. of fat.
By Storch's do	...	...	0.65	0.64 ditto

Below I give some examples showing how far I have found the results obtained by the Lactocrite to compare with chemical analysis.

Chemical analysis.		Lactocrite.			
3.73	3.74	3.7	3.75	3.75	
		3.8	3.8	3.85	
		3.8	3.8	3.82	
4.08	4.07	4.1	4.2	4.2	
3.86		3.8	4.0	3.9	3.9

At least equally good results have been obtained by Mr. John Sebelien, lecturer to the Agricultural College, Ultuna, Sweden, and superintendent of the Dairy Laboratory of the same place. From his report I quote:—

Chemical analysis.		Lactocrite.			
3.68		3.65	3.65	3.70	
		3.70	3.70	3.67	
		3.67	3.70	3.70	
2.76		2.77	2.80	2.77	
		2.80	2.80	2.75	
2.70		2.65	2.70	2.65	2.70

These samples, which are by no means picked, will show that the Lactocrite is able to give a very close estimation of the amount of fat in milk. I think it may fairly be claimed for the Lactocrite that it will give an estimation within 0.1 of the amount of fat in whole milk.

When skim-milk is treated in the Lactocrite the results will fall somewhat below those of the analysis, as seen in the following examples:

Chemical analysis.		Lactocrite.			
1.14	1.17	1.05	1.0		
		1.07	1.05		
0.87	0.90	0.75	0.8	0.75	0.8
		0.8	0.75	0.8	0.65
		0.82	0.75	0.8	0.8

Separated milk, from the cream separator, having but very little fat left in it, cannot be tested by the Lactocrite in the usual way, as many trials have shown the results to be about 0.2 per cent. too low, which difference in analysing separated milk of course cannot be allowed. Equally low results have been obtained from buttermilk.



Sour milk, even curdled, may be treated in the Lactocrite just as well as sweet milk, as the strong acetic acid will dissolve the casein of sour milk as easily as that of sweet milk. The only difficulty lays in the measuring off the 10 c.c. of a true average quality.

One great advantage of the Lactocrite is the very simple way in which it is worked, so that no skill is necessary, but any dairyman may obtain as good results as the apparatus is able to yield. In order to illustrate this I give below the results obtained by two persons at their first attempts; the first person is a dairyman used to heavy work. By way of a check I myself made some tests of the same milks:—

By myself.	Dairyman.		
3.1	3.1	3.2	3.2
3.2	Failed	3.2	3.2
3.3	3.1	3.3	3.3
2.65	2.65	2.6	2.6
2.65	2.65	2.6	2.65

These very favourable results are of importance as showing that in the Lactocrite is at last found the long wished-for apparatus, possessing the two qualities not hitherto combined—simplicity of construction and working and sufficient correctness for all practical purposes.

The Lactocrite will, no doubt, be found invaluable for butter dairies, or dairy factories buying milk from different farmers, by enabling them to carry out the system of paying for the milk according to the amount of butterfat which is the only fair system. At present, both in England and in other countries, the farmer whose milk will make butter at a rate of 3 lbs. per 100 lbs. of milk gets the same price as the farmers whose milk is so rich as to give 5 lbs. of butter per 100 lbs. of milk, which of course is most unfair. When milk is paid for according to the fat contained in it, the temptation to skim it is done away with, and besides, a great encouragement is given to the production of rich milk.

The Lactocrite will also prove of use for analysts who have access to a separator stand, as it will give in a short time a more exact determination of the amount of fat than any other apparatus. In this connection it will be of interest to know that a special construction of it has been adapted to fit Dr. de Laval's small Hand Separator, worked by hand and requiring no foundation.

#### DISCUSSION.

Dr. VIETH said he had listened with great interest to the papers, and he thought himself that instruments and methods for determining the most valuable constituent of milk—fat—which can be worked outside the chemical laboratory, and yet give correct results in the hands of non-chemists were of the utmost importance to dairy-farming and dairy-industry. From what he had heard that night, and learned previously, he had no doubt that the Lactocrite was a very useful apparatus, and that the results obtained were highly satisfactory. He was astonished to see so close an agreement with results relating to skim milk, because the inventor distinctly states that if skim milk is examined with the apparatus .2 per cent. must be added to the result in order to bring up the fat to the percentage actually present. With regard to the cooling down of the

test-tubes, while reading off the results, he did not think that a matter of great consequence, as the tubes were made of pretty thick glass. He might mention that the Lactocrite was not the only apparatus of its kind, but that there also exists a centrifugal control-apparatus in connection with the Danish cream separator. Each apparatus appears to have some merits of its own; but there was scarcely any occasion for a close comparison of the two, as they would never compete with each other. The Lactocrite cannot be used where the Danish separator is being worked, nor can the Danish control apparatus be employed in connection with the Swedish separator.

Mr. ADAMS was highly gratified to see the remarkable corroboration the Lactocrite afforded to his own method of analysis. He had been favoured by Mr. H. Faber with some particulars respecting the other mode of analysis employed by him, and referred to in his paper, also with specimens of the apparatus which Mr. Faber makes use of, consisting of a conical tube some 6 or 8 inches long, plugged at the bottom or smaller end with wool, through which a small glass tube passes, the space between it and the conical tube being filled with pumice stone broken to a coarse powder—which pumice takes the place of the sand, powdered glass, or plaster of Paris of other methods. The pumice is first ignited, then placed in a porcelain capsule, charged with the milk, which, when dried, is transferred to the tube to be extracted by ether under an upright condenser.

The speaker's experience of the process, as compared with his own, was that the yield of fat is 29 per cent. below truth, and besides its failure to extract the whole of the fat, he finds the pumice an unwieldy, disagreeable material to use as an absorbent, it being, in his opinion, much inferior to sand, glass or plaster, not only by reason of its ineffectiveness, but also because of its bulk and the difficulty of detachment from the porcelain capsule without loss of substance, and the destructive action it has on the capsule by scratching of the enamel.

#### NOTE ON REICHERT'S DISTILLATION PROCESS.

By ALFRED H. ALLEN.

*Read at the Meeting, December 8th, 1886.*

In a paper read before this Society in the spring of last year, and published in the ANALYST for June, 1885, I described my experience of Reichert's method of assaying butter-fat, and gave a number of figures illustrating the results obtained with oils of various kinds. These figures professed to represent the volatile acids in terms of butyric acid, and were obtained by multiplying the number of centimetres of decinormal alkali required for neutralising the distillate from 2.5 grammes of fat by the factor 0.312. I am indebted to Mr. W. F. K. Stock for pointing out an error in this factor, which should be 0.352.\* As the erroneous factor was used in calculating the figures already referred to, it follows that they are all too low in the proportion of 312:352, or 39:44.

A further experience in the employment of Reichert's process for examining fats has led me to abandon the expression of the results in terms of butyric acid, in favour of a statement of the weight of caustic potash neutralised by the distillate from 100 grammes

\* 1 c.c. of  $\frac{N}{10}$  alkali neutralises 0.0088 gramme of butyric acid, and  $\frac{0.0088 \times 100}{2.5} = .352$ .

The erroneous factor may have resulted from the fraction  $\frac{0.0078 \times 100}{2.5} = .312$ .

of the oil. This is obtainable by multiplying the volume of decinormal alkali neutralised by the distillate from 2·5 grammes by the factor 0·2244.\*

The following table contains a number of results expressed in both ways:—

Fatty Oil.	C.C. of $\frac{N}{10}$ alkali required by 2·5 gr.	KHO required by 100 parts of oil.	Observer.
Butter or Milk-fat ; Cow's ..	12·5 to 15·2	2·80 to 3·41	Reichert, Caldwell, Moore, Allen, etc.
„ „ Ewe's ..	13·7	3·07	Schmitt
„ „ Goat's ..	13·6	3·05	„
„ „ Porpoise's ..	11·3	2·51	Allen
Cocoonut oil .. .. .	3·5 to 3·7	0·78 to 0·83	Reichert, Moore, Allen
Palmtree oil .. .. .	2·4	0·54	Allen
Palm oil .. .. .	0·8	0·18	Moore
Cacao butter .. .. .	1·6	0·36	„
Butterine and oleomargarine ..	0·2 to 1·6	0·04 to 0·36	Caldwell, Moore, Allen
Whale oil .. .. .	3·7	0·83	Allen
„ .. .. .	12·5	2·80	„
Porpoise oil .. .. .	11 to 12	2·47 to 2·69	„
Sperm oil .. .. .	1·3	0·29	„
Bottlenose oil .. .. .	1·4	0·31	„
Menhaden oil .. .. .	1·2	0·27	„
Codliver oil .. .. .	1·1 to 2·1	0·24 to 0·47	„
Sesamé oil .. .. .	2·2	0·48	„
Cottonseed oil .. .. .	0·3	0·07	Moore
Castor oil .. .. .	1·4	0·31	Allen

From these results, it is evident that the fats of different kinds of milk (butter-fats) are sharply distinguished from nearly all other fats by the large proportion of soluble volatile fatty acids they yield by Reichert's process. The most remarkable exception is presented by porpoise oil and some samples of whale oil. In porpoise oil I have found 5 per cent. of valeric acid, and Chevreul obtained as much as 9·63 per cent. In a recent paper I pointed out that in porpoise-butter the glyceride of valeric acid appeared to replace the butyryn characteristic of the butter of terrestrial mammals.

Some of the chemists who have employed Reichert's process take the precaution to filter the distillate before titrating it, so as to get rid of any volatile acids which may be insoluble or very sparingly soluble in water. This plan may sometimes be adopted with great advantage. Thus when the solution of the soap obtained by saponifying cocoonut or palmtree oil is acidulated and distilled, a notable proportion of lauric acid passes over and solidifies in the condenser or on the surface of the distillate; and by adding water to the contents of the retort, again distilling, and repeating this process several times, a very considerable proportion of volatile fatty acids can be obtained from cocoonut oil. In assaying butter the appearance of insoluble acids in the distillate would

\* 1 c.c. of  $\frac{N}{10}$  alkali contains 0·00561 gramme of KHO; and  $\frac{0·00561 \times 100}{2·5} = 0·2244$ .



furnish a valuable indication of the presence of cocoanut oil, and they should be removed by filtration; or the distillate will be found to neutralise so large a volume of alkali as considerably to diminish the practical value of the process as a means of distinguishing butter from butter-substitutes, as has been pointed out by Moore and others. Latterly, I have adopted the plan of filtering the distillate in all cases, washing the filter with cold water, and then immersing the filter, with any adhering insoluble acids, in alcohol, which is then titrated with decinormal alkali and phenolphthalein. In the case of ordinary butters and butter-substitutes the insoluble volatile acids only neutralise about 0.2 c.c. of decinormal alkali.

The question having recently been raised, the following experiments were made at my request by Mr. William Barracough on a sample of butter-fat, in order to ascertain the variation in the results of Reichert's process produced by modifications in the methods of conducting the saponification and distillation.

1. 2.5 c.c. of butter-fat was saponified by alcoholic potash in an open basin, the alcohol evaporated off completely at a steam heat, the residual soap dissolved in water, the solution acidulated with sulphuric acid in slight excess, diluted to 75 c.c., and distilled gently in a globular flask with side-tubulure adapted to a condenser, until 50 c.c. had passed over. The flask held 460 c.c. up to the side-tube, and some fragments of pumice-stone coiled round with platinum wire were added to the contents, to promote evolution of vapour.

2. An exact repetition of No. 1 experiment.

3. Saponification was effected in a flask furnished with a long tube and heated by steam. The subsequent manipulations were the same as in experiment 1.

4. Saponification was effected in a well-closed bottle placed in the water-oven. Other manipulations unchanged.

5. Manipulation exactly as in experiment 3, except that the distillation was conducted in a flask fitted to the condenser by a cork and bent tube.

6. Conducted as in experiment 3, except that the distillation was conducted in a retort.

7. Blank experiment with the alcoholic potash employed in the previous experiments, the manipulation being like that in experiment 3. The alcoholic potash was brown and not very recently prepared.

#### Experiments.

Decinormal Alkali for 2.5 grammes.				Decinormal Alkali for 2.5 grammes.			
No. 1	..	..	11.80 c.c.	No. 5	..	..	12.40 c.c.
No. 2	..	..	11.85 "	No. 6	..	..	12.45 "
No. 3	..	..	12.40 "	No. 7	..	..	0.25 "
No. 4	..	..	12.50 "				

These results show that a sensible loss occurs if the saponification be conducted in an open basin, doubtless owing to the formation of butyric ether. On the other hand, the exact nature of the distilling apparatus appears to be of little importance. This latter conclusion is not in accordance with the experience of some other chemists.

*Conclusion of the Society's Proceedings.*

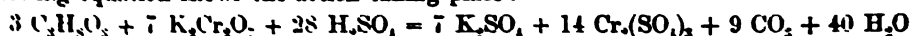
## A NEW PROCESS OF ESTIMATING GLYCERIN IN FERMENTED LIQUIDS.

By L. LEGLER.\*

1. THE process for the determination of glycerin in wines, as decided on by the Berlin Committee, has of late been improved, but there are still great sources of error, chiefly the loss of glycerin in the process of its purification by ether-alcohol. Pure glycerin is certainly soluble in a mixture of 1 volume of alcohol and 2 volumes of ether, but when other insoluble bodies are present they retain a not inconsiderable amount of glycerin, sometimes as much as 20 per cent. of the total. Repeated treatment with the ether-alcohol fails to completely remove all the glycerin. The process which I now recommend is based on the following principle:—

The carbon of glycerin may be readily oxidized to carbonic acid by means of sulphuric acid and potassic dichromate. By using a Wills carbonic acid apparatus, and estimating the loss in weight, we can calculate the amount of glycerin.

The one flask contains the glycerin mixed with a saturated solution of potassic dichromate, the other contains, as usual, strong sulphuric acid. After the apparatus has been weighed, a little air is drawn out, which causes some of the acid to mix with the chromate. A regular evolution of carbonic acid soon sets in, but must be assisted towards the last by gently boiling. The flask containing the sulphuric acid must be kept cool. When no more gas bubbles are formed, the apparatus is cooled by partial immersion in cold water, and the remaining carbonic acid is expelled by a current of dry air. The apparatus is now reweighed, and the loss represents carbonic acid. The following equation shows the action taking place:—



One part of glycerin therefore requires about 7.5 parts of  $\text{K}_2\text{Cr}_2\text{O}_7$  and 10 parts of  $\text{H}_2\text{SO}_4$ , but an excess of each is of course used. The dichromate must be purified by recrystallization.

In using a delicate balance I have a smaller apparatus made on purpose, and can then operate on .25 gramme of glycerin, but I also can work on .75 or 1 gramme; if the balance is still sensitive to .01 or .005 grammes. The operation takes about one hour for every .25 gramme of glycerin. The following test analyses show the accuracy of the method. The glycerin actually weighed had a specific gravity of 1.2339 at 15°C.; and according to Lens's tables contained 86.8 per cent. of pure glycerin.

Pure glycerin taken.	Carbonic anhydride.	Glycerin found.
1.00 grammes. . .	1.445 grammes. . .	1.007 grammes.
.500 " " "	.725 " " "	.505 " "
.500 " " "	.710 " " "	.495 " "
.250 " " "	.350 " " "	.244 " "
.248 " " "	.3562 " " "	.2483 " "
.250 " " "	.3610 " " "	.2515 " "
.248 " " "	.3587 " " "	.2500 " "

The last three analyses were done with the aid of a delicate balance.

## 2. Estimation of glycerin in wine:—

The crude glycerin obtained from 100 c.c. of wine, after evaporation with 3 c.c. milk of lime and 2 grammes of quartz, and extracting the mass with alcohol of 96 per

\* *Reper. Anal. Chemie*, 47.

cent., is, after weighing, diluted up to a definite bulk, and aliquot parts are taken for the ash and the oxidation process. A white wine, containing 8.54 per cent. of alcohol and 2.07 per cent. solid matter, gave in 100 c.c. 1.4 grammes crude glycerin with .1278 ash.

25 c.c. of the glycerin diluted up to 50 c.c., yielding .725 grammes  $\text{CO}_2$  = 1.10 per cent. of glycerin. A duplicate experiment gave 1.47 crude glycerin with .136 ash, .710  $\text{CO}_2$  = .99 per cent. glycerin; the average thus being 1 per cent., and the relation between alcohol and glycerin as 100 : 11.7.

### 3. Estimation of glycerin in wine after it has been purposely added.

Three lots of 100 c.c. each of the same wine were mixed respectively with .125, .250, .500 grammes of glycerin, and analysed as before. The results were as follows:—

Crude glycerin.	Ash.	$\text{CO}_2$ (25 c.c. from 50 c.c.)	Glycerin in 100 c.c.
1.55 .. ..	.1496 .. ..	.80 .. ..	1.115
1.75 .. ..	.1400 .. ..	.90 .. ..	1.254
2.07 .. ..	.1172 .. ..	1.07 .. ..	1.492

Allowing for the 1 per cent. of natural glycerin in the sample, we obtain .115, .254, and .492 per cent. of glycerin.

The chromate solution from the two last experiments was afterwards submitted to distillation, and yielded about .15 gm. of acetic acid, derived from impurities in the crude glycerin.

4. Suppose it could be taken for certain, the process removes such bodies as sugar, tartaric, malic, citric, and tannic acids, which would interfere with the accuracy of the oxidation process, it would still be necessary to ascertain if there are other bodies in the crude glycerin which would yield carbonic acid. From the experiments it would appear such a quantity of carbonic acid is constant. To separate any of these bodies, the diluted crude glycerin was mixed with an ammoniacal solution of lead acetate, the precipitate filtered off, washed, dissolved in acetic acid, and this fluid, after heating to expel  $\text{CO}_2$ , submitted to the oxidation process.

A precipitate obtained from 100 c.c. of wine yielded an amount of carbonic acid equivalent to .035 gm. of glycerin. The true amount in the sample was therefore  $1.00 - .035 = .965$  per cent.

### 5. Comparison of the old process with the oxidation process.

100 c.c. of the same wine treated by the old process gave the following weights of glycerin:—

After the first extraction	.7177	including	.015	ash.
"    second    "	.1488	"	.0123	"
The residue weighed	.3819	"	.096	"

The oxidation of these residues gave an amount of carbonic acid corresponding with .6300, .1295, and .1662, total .9257 grms. of glycerin, from which must be deducted .035. From these figures it will be seen the oxidation process is the best, the results obtained by the old process being too high, on account of small quantities of fatty and nitrogenous matters, and of organic acids, which cause the ash to contain potassic carbonate. The total glycerin, .9257, differs .0383 from the .965 gm. previously found, and this difference is due to loss during the drying of the glycerin after purifying with ether-alcohol.



I have also got promising results with beer and sweet wines. 100 c.c. of Lager beer with 5.5 per cent. of extract, yielded after evaporation with 8 c.c. milk of lime and 5 grm. of quartz, .069 grm. of glycerin. The same beer mixed with .25 grm. glycerin gave .308 grm. The quantity of recovered glycerin was therefore  $.308 - .069 = .239$  grm.

For sweet wines I have introduced a modification based on the fact that glucose or invert-sugar are completely decomposed by boiling with strong alkaline fluids, like baryta water, and forms salts insoluble in spirits of wine.

A quantity of wine as will yield about 3 grammes of solid residue is evaporated, first in a large basin, finally in a smaller one, with excess of baryta water, containing about 7.8 grammes of  $Ba_2HO$ . Calcium hydrate does not answer so well, as too much of it is required, and the bulk is inconveniently increased. When no more water vapour is given off, the mass is mixed with 8 grammes of quartz, and then extracted four times in succession with 80 c.c. of alcohol of 85 per cent. The mixed alcoholic extracts are distilled, and the residue is then ready for the oxidation process. If the wine contained cane-sugar, this must first be inverted by boiling with  $HCl$ .

To test the accuracy of this process, I separated any barium from the fluid, by passing a current of  $CO_2$  and adding a few drops of ammonia. After filtering and evaporating to a syrup, the residue, when dissolved in water, gave but a faint precipitate with ammoniacal solution of lead acetate. Both filtrate and precipitate were subjected to the oxidation process. A sweet wine, with 15.17 per cent. alcohol and 15.91 per cent. extract was analysed, with the following result—

1. 20 c.c. = .320  $CO_2$  = .223 glycerin.

2. 20 c.c. + .496 added glycerin = 1.02  $CO_2$  = .711 glycerin.

Glycerin recovered,  $.711 - .223 = .488$ .

When *first* treated with ammoniacal lead acetate, precipitates were formed, which yielded an amount of  $CO_2$  corresponding with .0113 and .0146 grammes of glycerin. I hope to soon publish the results of further experiments.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

A NEW TEST FOR PICRIC ACID AND BINITROCRESOL. K. FLECK. Repert. Anal. Chem., 1886, No. 48.—As many makers of farinaceous foods are in the habit of colouring them, it has been thought they may occasionally use picric acid. This suspicion is, perhaps, not well founded, as picric acid would communicate a bitter taste, and so render the products unsaleable. As it is, however, possible picric acid (Welter's Bitter) may be unintentionally substituted for binitrocresol (Victoria yellow), which is largely used in the trade, it becomes important for an analyst to be able to positively distinguish these two colouring matters, which hitherto has only been possible by tasting the alcoholic extract.

If a solution of picric acid is concentrated in a small porcelain dish, and mixed with

a few c.c. of 10 per cent. hydrochloric acid, the colour is at once destroyed. Binitro-cresol is also decolourized after a few minutes. If now a piece of zinc is introduced, and allowed to act for one or two hours, the picric acid turns a fine blue; but binitro-cresol turns blood red. To apply the reaction to foods, they must be powdered and extracted with alcohol. The residue obtained after evaporating off the spirit must be carefully tasted for bitter, and then treated with hydrochloric acid and zinc as described.

L. DE K.

### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ESTIMATION OF THE DECOLOURISING POWER OF ANIMAL CHARCOAL. G. LAUBE. Repert. Anal. Chem., 1886, No. 49.—Analysts, unless engaged in sugar refineries, are not often called upon to perform this analysis, and therefore do not as a rule possess the required costly apparatus. The author therefore thinks an easy and practical process will be of some service to them. As a rule the question is merely whether a char has already been used or not.

The first thing is to select a sample of undoubtedly genuine char, to remove any whitish or suspicious-looking particles, then to powder and pass through a sieve. The powder is dried at 110° C., and kept in a stoppered bottle. It is labelled *Standard Animal Charcoal*.

A solution of caramel is now prepared. 100 grammes of the commercial article, which has about the thickness of honey, are dissolved in 100 c.c. of water, then mixed with 100 c.c. of alcohol, and diluted with water up to one litre. After standing for a day or two, it must be filtered. It is labelled *Standard Colouring*.

The decolourizing power (=100) of the standard charcoal is now determined as follows:—Five grammes are put into a not too small flask, mixed with 200 c.c. of water, and heated to boiling. 10 c.c. of the standard colour are now added, and the whole again boiled for ten minutes, under an upright condenser, to avoid loss by evaporation.

The fluid is now filtered through a double filter; the filtrate, which must be quite clear, is, after cooling, mixed with a drop of solution of caustic soda, and put into a cylindrical glass.

200 c.c. of water, and a drop of caustic soda, are now put into another cylinder of exactly the same diameter, and as much standard colour is now added as will give it the same depth of colour as the filtrate from the char. Suppose 2.1 c.c. were required, the char would have absorbed  $10 - 2.1 = 7.9$  c.c. standard colouring. If now a suspected sample should, when similarly treated, absorb  $10 - 4.5 = 5.5$  c.c. standard colour, its decolourizing coefficient would be—

$$\frac{5.5 \times 100}{7.9} = 70.$$

As the decolourizing power also depends in a slight degree on the state of division, the same sieve must be used.

L. DE K.



## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

To the Editor of the ANALYST.

SIR,—I find in the last number of the ANALYST, in the report of the discussion on my paper, some remarks by Dr. Alder Wright on the plummet-method of taking specific gravities. I answered his criticisms on the occasion of the meeting, and understood that such parts of the discussion as would appear in the ANALYST were all replied to in the abstract of my remarks published on page 230. This, however, is not the case, as no reply appears to Dr. Wright's criticisms. I shall therefore be obliged if you will allow me space for the following comments.

In the first place there is a distinct practical advantage in comparing with water at the ordinary temperature taken as unity the figures obtained with oils at the boiling-point of water. This is in consequence of the construction of the Westphal Balance, and similar pieces of apparatus, and the weights used therewith. Practically, the numbers I give as expressing the densities of oils are the relative weights necessary to be used to counterpoise a plummet which is counterpoised by 1 gramme when immersed in distilled water at 15° C. If the figures which were the actual result of observation were to be compared with water at the boiling-point as a standard, a troublesome calculation would be necessary, and, as Dr. Wright points out, the results would be vitiated to a certain extent by well-known causes.

But I would point out that the value of the figures as accurate expressions of the relative weights of equal measures of liquids at the boiling-point of water, compared with that of an equal measure of water at the ordinary temperature, is a very different thing from the constancy of the figures obtained. Thus, it may not be a fact that a cubic centimetre of butter-fat at 99° C. weighs .8680 gramme, but it is a fact that a particular sample of butter-fat which gives an indicated density of .8680 at 99° C. by the Westphal balance will, on repeating the experiment, give a figure not varying by more than .0002 from that first obtained. Practically, what we desire to know is the indicated comparative density under the conditions in question. In the present condition of chemistry there is absolutely no useful deduction to be drawn from the density of a liquid fat, except in comparison with the density of other fats; and if the observations be so made as to render all the results comparative, every object having either a theoretical or practical interest is attained. If any one have any desire for a more complicated mode of expression by all means let him adopt it, but I feel sure that analysts generally will prefer to employ figures obtained by directly reading off the indications of the balance, than certain other figures derived from the last by a somewhat complicated series of considerations, and which, in case of necessity, it would be perfectly impossible to get the average magisterial or legal mind to follow.

I fully agree with Dr. Wright in his objection to such expressions as "specific gravity at 100° compared with water at 15°," and have indicated my views pretty strongly elsewhere ("Commercial Organic Analysis," vol. 1., page 7). The difficulty is to find some short mode of expression which would have a chance of being generally adopted by chemists, and which would express what was really meant. As pointed out by Dr. Wright, owing to the expansion of the plummet, the actual weight indicated would not be accurately the weight in grammes of 1 cubic centimetre at the boiling-point. In default of a better, I would suggest the term "*indicated plummet-gravity*" for expressing densities indicated by the plummet method, and would add the temperature of the liquid in each case; it being understood that the plummet is one which indicates unity when immersed in water at 15° C. Thus the "*indicated plummet-gravity*" of butter-fat at 99° C. ranges from .8670 to .8700, with an average of .8680; while butterine usually indicates between .8585 and .8625 under similar conditions.

—I am, sir, yours truly,

ALFRED H. ALLEN.

1, Surrey Street, Sheffield, December 21st, 1886.

"ADDING SALT TO MILK."

To the Editor of the ANALYST.

DEAR SIR,—With reference to the footnote on the above case, this is not the first conviction in Scotland for a similar offence. I enclose a newspaper report of the case which was tried on March 12th, 1876. Kindly return the inclosure, and make the correction in your next issue.—Yours truly,

85, Raglan Road, Smethwick, near Birmingham, 16th December, 1886.

W. McCOWAN.

BOOKS, &c., RECEIVED.

AMERICAN Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; British and Colonial Druggist; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Hospital Gazette; Illustrated Science Monthly; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Manual for the use of the Board of Health of Massachusetts; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Outlines of Quantitative Analysis, by A. Humboldt Sexton; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Sanitary Examination of Water, Air, and Food, by Cornelius B. Fox, M.D.; Scientific American; Society of Arts Journal.

NOTICES TO CORRESPONDENTS.

ALL Communications to be addressed to 325, Kennington Road, London, S.E.

# THE ANALYST.

FEBRUARY, 1887.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Annual General Meeting of the Society was held at Burlington House on Wednesday, the 12th ult. In consequence of illness, Dr. A. Hill, the retiring President, was unable to be present, and the chair was taken by Dr. Muter, one of the Vice-Presidents.

The minutes of the previous meeting were read and confirmed.

The balance-sheet for 1886 was read by Mr. DYER, who announced that Dr. Muter was about to pay over to the Society the sum of £9, that being the amount left over after paying all expenses in connection with THE ANALYST during 1886, in addition to the £5 which he had paid over for 1885.

The balance-sheet was then received and approved, and ordered to be printed and circulated, as usual.

Mr. DYER read a letter from the retiring President. Dr. Hill stated that he was confined to his bed with bronchitis, and expressed his great regret that he was unable to be present to welcome the new President, and to deliver his retiring address, which was then read by Mr. DYER as follows:—

GENTLEMEN,

The time has arrived for my putting off both the honours and responsibilities of the office of your President, and, in occupying this chair for the last time, I take the opportunity of saying a few words of acknowledgment and congratulation—acknowledgment of the support, assistance, and kindness which, during my two years' tenure of office, you have uniformly extended to me; and congratulation on the continued success and prosperity of the Society.

This success is shown partly by the increase of members and associates, from 173 in the year 1884, and 181 in the year 1885, to 187 in that of 1886, as shown more in detail in the following statement:—

	1884	1885	1886
Honorary members .. .. .	10	10	10
Ordinary members .. .. .	142	149	152
Associates .. .. .	21	22	25

Ten ordinary members and four associates have been elected; six ordinary members and one associate have resigned, while one member, Mr. H. Sugden Evans, of the Inland Revenue Department of Ottawa, Canada, has died.

The Balance Sheet, that you have heard read, is the most satisfactory that the Society has yet known. We have nearly £100 in hand, and this, in face of the fact that we have a long list of subscriptions in arrear, which, the secretaries inform us, must, they think, be attributed, in some degree, to "bad times," as more effort than usual has been made, during the past year, to keep down this item in the accounts. The Society has been indebted, during the past year, to Dr. Muter, for a donation of £5, and I have great pleasure in announcing that he has this evening promised us a further donation of £9.



As evidence of the sustained interest and activity of the Society, I may state that during the year, twenty-three papers were read, as compared with twenty-one in 1885. Eight of them, or more than one third of the whole number, referred to the always important subject of milk, three to water, three to oils, two to soap, and one each to bread, butter, cheese, pepper, starch, and waxes and allied bodies; and finally there was one upon Fuller's Slide Rule, for facilitating chemical calculations.

One event of the year, worthy of special mention, is the adjourned discussion on the Report of the Milk Committee, brought up last year, resulting in the adoption of the report of that committee, after an adjournment of three months, in order to allow all chemists an opportunity of testing the value and accuracy of the Committee's results. It was, at the same time, recommended that all analysts should use the process (a modification of Mr. Adams'), in preference to any other. It was very gratifying to find that members, who, at first, had hesitation or doubts about the advantages of the process, became fully convinced of their reality, after a sufficient trial of it. I believe that the conclusion arrived at, with only one dissentient voice, will be fully justified in the future; indeed, it has recently received valuable corroboration from the experiments made in the Hanover State Laboratory, with a view to test the respective merits of the Lacto-butyrometer method, Bell's method, Soxhlet's areometric method, and Adams' method. Dr. Baertling sums up the work (an account of which, taken from the "*Reperitorium f. Anal. Chem.*," is contained in *THE ANALYST* for last November), by a report strongly in favour of Adams' method, on the very sufficient grounds of greater expedition, simplicity, economy, and accuracy; further, especially on account of its suitability for sour milks, giving the results of analyses of the same milks, both in the fresh and sour condition, and showing them to be practically identical; and, in conclusion, recommending the results as a legal standard in Germany.

Such testimony as this, coming from a tribunal equally competent and impartial, cannot fail to be most acceptable to the Milk Committee, the Society, and Analysts in general.

The influence of our Society has been, during the whole time of its existence, exerted in the same beneficial manner in other directions, notably in the processes of analyses in connection with butter, bread, and other important articles of food and drink, resulting in the elaboration of many perfected out of crude processes, diminished disagreement among analysts, greater certainty of success in legal prosecutions, greater fairness to buyer and seller, and lastly, the practical advantage of progressive diminution of adulteration—the end and aim of the Act. This successful outcome of the operation of the Sale of Food and Drugs Act is evidenced by the Annual Report of the Local Government Board, recently published, in which it is stated that at the time of the inquiry instituted by the *Lancet* Sanitary Commission in 1854-1856, more than 50 per cent. of the samples analysed were "reported against." In the year 1877, the percentage of adulteration in articles examined was 19·2, and the average percentage for the years 1877-1881 was 16·2; in 1884 it fell to 14·4; while in 1885 it was found to have fallen to 13·2, which, I believe, is the lowest point ever attained. It is a somewhat remarkable coincidence, and, perhaps, a satisfactory indication of a general improvement, that the percentage of adulteration in the Borough of Birmingham in 1885 in 914 samples was exactly 13.



I cannot omit a reference to the country meeting of 1885, inasmuch as it proved to be a source of both profit and pleasure to all those members and their friends who were fortunate enough to participate in it. Through the courtesy and valuable assistance of Mr. Adams, many of the various attractions of Maidstone and its environs were revealed. Several large paper-mills were visited, and the very interesting process of paper-making examined in all its stages. The extensive and modern processes and appliances of a local brewery were also inspected in great detail; while, to conclude and crown all, a most agreeable trip was made up the Medway, on the kind and hospitable invitation of Mr. Adams.

Several of the party subsequently visited Canterbury, where, under Mr. Harvey's excellent guidance, they were shown the principal archaeological and ecclesiastical monuments of the city, and the new waterworks, where the softening of the water supply from the chalk is carried on.

In conclusion, I congratulate the Society that the presidential mantle is about to fall on such worthy shoulders as those of my successor. The Society, in electing Mr. Allen to the presidency, is doing honour both to itself and Mr. Allen, a gentleman who, by his ability and acquirements, his great practical experience, his excellent analytical work, and his literary productions, possesses a claim to the distinction, which it is a pleasure to recognise, and qualities which cannot fail to act advantageously on the interests of the Society which, I sincerely trust, has before it a long career of constantly increasing usefulness and prosperity.

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Dr. MUTER said they had listened to the address with great interest, and it needed no words of his to insure their passing a hearty vote of thanks to Dr. Hill for his conduct in the chair during his Presidency. Dr. Hill was one of the country members, and it was often a great inconvenience for those gentlemen to attend in London, but he had been present at nearly every meeting during the time he was President. He had done his duty with great thoroughness, and had conducted their meetings well, and in an orderly manner, and kept up the respect due to the chair.

Dr. SEATON seconded the vote of thanks, and said that Dr. Hill was an old Medical Officer of Health. He was the first of the country members to be appointed to the chair of the Public Analysts' Society, and now the Medical Officers of Health Society had followed their example, and had lately elected him President of that Society.

The vote of thanks was carried unanimously.

Dr. BOSTOCK HILL, in returning thanks on behalf of his father, said that Dr. Hill had taken a cold while attending to his official duties, and going out in the late rough weather had made him worse, so that on the 10th he was obliged to take to his bed. He had prepared his address while in considerable pain, and labouring under great difficulty of breathing. Among other reasons why he specially wished to attend was that he felt he ought to be present to welcome the new President, and it was a matter of very great regret to him that he could not be there to do so.

Dr. STOKES proposed, and Mr. ASHBY seconded, a vote of thanks to the Vice-Presidents and Council of the Society.

Mr. HEISCH proposed, and Mr. S. HARVEY seconded, a vote of thanks to the Treasurer and Hon. Secretaries.

Dr. MUTER proposed a vote of thanks to the Chemical Society for the use of their rooms, and the Secretaries were requested to communicate the same to the Council of that Society.

Dr. Bostock Hill and Mr. Johnstone having opened the ballot papers, Dr. MUTER announced that the following gentlemen had been elected as Officers and Council for the ensuing year:—

*President*.—A. H. Allen, F.C.S., F.I.C.

*Vice-Presidents* (who have filled the office of President).—A. Dupré, Ph.D., F.R.S., F.C.S., F.I.C.; C. Heisch, F.C.S., F.I.C.; Alfred Hill, M.D., F.C.S., F.I.C.; J. Muter, Ph.D., M.A., F.C.S., F.I.C. (Who have not filled the office of President).—J. Baynes, F.C.S., F.I.C.; P. Vieth, Ph.D., F.C.S.; C. R. Alder Wright, D.Sc., F.R.S., F.C.S., F.I.C.

*Treasurer*.—C. W. Heaton, F.C.S., F.I.C.

*Hon. Secretaries*.—Bernard Dyer, B.Sc., F.C.S., F.I.C.; Otto Hehner, F.C.S., F.I.C.

*Other Members of Council*.—M. A. Adams, F.R.C.S., F.I.C.; W. Fox, F.C.S.; C. N. Hake, F.I.C.; C. T. Kingzett, F.C.S., F.I.C.; T. Boverton Redwood, F.C.S., F.I.C.; W. F. K. Stock, F.C.S., F.I.C.; R. R. Tatlock, F.R.S.E., F.C.S., F.I.C.

The names of those Members of Council whose term of office has not yet expired, and who consequently do not retire this year, are A. Wynter Blyth, M.R.C.S., F.C.S., F.I.C.; A. Bostock Hill, M.D., F.I.C.; R. H. Harland, F.C.S., F.I.C.; S. Harvey, F.C.S.; and E. Seaton, M.D., F.R.C.P.

The following gentlemen were also elected:—

As Member: Mr. Harald Faber.

As Associate: Mr. Adolphus Dresel, assistant to Mr. Johnstone.

Mr. A. H. ALLEN, the newly-elected President, then took the chair amidst loud applause, and said:—

I have to thank you, gentlemen, for the very hearty manner in which you have just expressed yourselves, and I trust I may take it as an earnest of the success I hope to meet with during my time of service. I have the interests of the Society very much at heart, and shall certainly do all in my power to advance them, and to render the Society even more successful in the future than it has been in the past, and I have great pleasure in thinking I shall have, not only the assistance of the Secretaries, but of all the other members of the Council. From the balance-sheet just read we see we are in a very good financial position, and there certainly does not seem to be any want of interest in the proceedings of the Society, or any falling off the members. The proceedings of the Society are becoming of more general interest, and, in fact, the Society has now established itself as it ought to be. It is in its teens, and I hope to see it yet more flourishing. We can never expect to be a large Society, but we may fairly hope to see it increase in usefulness, and I shall certainly do all I can to advance its interests in every possible way.

The following papers were then read and discussed:—



Arsenical Glauber Salts: Its Toxic Effects, by Sir C. A. Cameron, M.D., R.C.S.I.

On the Estimation of Methylalcohol, by Otto Hehner, F.C.S., F.I.C.

On a New Pepper Adulterant, by Dr. Campbell Brown.

Dr. Campbell Brown's paper was illustrated by a specimen of *Poivrette* exhibited under the microscope, by Dr. Muter on his behalf, and it was proposed that the Editor of THE ANALYST should make arrangements to circulate slides of this adulteration to the members. Full particulars of these arrangements will be found in the present issue.

After the meeting the annual dinner took place at the Criterion, Piccadilly, where the members and several friends spent a very enjoyable and convivial evening.

The next meeting of the Society will be held at Burlington House on Wednesday, the 9th February.

#### ON POIVRETTE.

BY PROF. J. CAMPBELL BROWN, D.Sc.

*Read at the Meeting, Jan. 12th, 1887.*

THE substance known in the pepper trade as "*Poivrette*," or "*Pepperette*," is now so frequently used for the purpose of "fraudulently increasing the weight and bulk" of commercial pepper, that the members of this Society ought never to omit a careful search for it in all samples of pepper officially submitted to them. As many commercial analysts do not appear to be yet familiar with *poivrette*, and as some public analysts have applied to me for specimens, a short account of it may be of use to the Society. It made its first appearance in Liverpool last summer, when more than one wholesale pepper-merchant brought me samples, and inquired what the substance was, and what were its properties. During the last three months I have met with it in between twenty and thirty retail samples of pepper.

*Poivrette* is a pale, slightly buff, or cream-coloured powder, resembling in the bulk the principal middle layers of the pepper-berry, when ground; and when mixed with pepper cannot be distinguished by the eye, nor even by the hand-lens, from particles of pepper. In the earlier samples the coarser particles could be isolated by spreading the pepper on a stiff sheet of paper held in a nearly, but not quite horizontal position; on tapping this with the finger tips, so as to make the larger particles jump gradually to the lower edge of the sheet, the *poivrette* particles could then be picked out, and easily distinguished from pepper by crushing them between the teeth. Recently, however, it has been so finely ground and sifted that it cannot always be partly separated in this way, although the toughness and hardness of the particles can always be distinguished by the teeth in a mixture.

Microscopic examination, with a  $\frac{1}{8}$ th or  $\frac{1}{4}$ th objective, shows that it consists of pale dense ligneous cells, some entire and marked with linear air spaces, some torn and indistinct.

The following letters (which afterwards appeared in some local newspapers) indicate the country from which it comes:—

"The following letter from Leghorn has been received by a local spice house, and similar letters have been circulated throughout the country :—

"LIVORNO, August 1, 1886.

"Dear Sirs,—I send you by this post two samples of an article called 'pepperette' (white and black), which is made of the pulp of a fruit growing in this country, which has the power of retaining the piquancy of pepper when it has been mixed with the same in the proper proportion. This is warranted to consist of this purely vegetable substance, and to contain nothing deleterious, consequently to be in no way detrimental to the health. The price is £8 per ton of 1,000 kilogrammes, goods delivered c.i.f. in Liverpool, packed in 2 cwt. bags; bags free, no tare, shipping weight;  $2\frac{1}{2}$  per cent. discount for cash. I export my pepperette very largely all over the Continent and to Great Britain, where, on account of its cheapness, it is used very much for blending pepper, which is sold as "prepared pepper," or "pepper not warranted genuine," in the same way as is done with mustard, or with ground coffee and chicory (the so-called French coffee). If so desired, the white pepperette can be had much lighter. If you desire any references I shall be happy to furnish you any amount in England, as well as on the Continent.—Yours truly

"A reply was forwarded, in due course, to the manufacturers of "pepperette," asking for further particulars and references, and the following letter was received :—

"Dear Sirs,—I am favoured with your letter, 16th instant, and note contents, "Pepperette." What you ask me is a question that is very frequently asked me by English houses, but I am always in the impossibility to reply to it; in fact, I *must* not do it. When I sell my "pepperette" (or "poivrette") to a firm, I bind myself not to mention their name to anybody, and will do so with your good selves, if I have the pleasure of being favoured with your orders. I make it a point of the question of secrecy with all my customers for this article, and cannot make an exception with you. Give me a sample order of a few tons, and I shall execute it to your entire satisfaction; payment after receipt and approval of the goods. However, for your guidance, and according to what I promised with my letter of the 13th inst., I now beg to subjoin a few English references, who can inform you concerning my respectability, but kindly do not mention to them anything about "poivrette," the same being houses from whom I import English goods (i.e., my firm, — — —). As already written, I shall be able to send sample of white poivrette of lighter colour by October next. In the meantime I trust to be favoured with your esteemed orders, and remain, dear sir,

"— — — — —"

I therefore examined, amongst other substances, walnut-shells, almond-shells and olive-stones. The cells of walnut-shells are dotted, though otherwise similar to poivrette; the almond-shells greatly resemble poivrette, and olive-stones still more closely resemble it. Chemical analysis indicates the closest correspondence between poivrette and olive-stones, as the following figures show :—

	Ash.	Matters soluble by boiling in dil. acid.	Albuminous and other matters soluble in alkali.	Woody fibre, insoluble in acid and alkali.	Starch.
White pepperette .. ..	1.33	38.32	14.08	48.48	None
Black pepperette .. ..	2.47	34.55	17.66	47.69	"
Ground almond-shells .. ..	2.05	23.53	24.79	51.68	"
Ground olive-stones .. ..	1.61	39.08	15.04	45.38	"



The stones of olives, imported in pickle for table use, gave 3.68 per cent. of ash, but well washed olive-stones, thoroughly burnt to a white ash, gave under two per cent. of ash-like poivrette. "White poivrette" is therefore cleaned very pale, and perhaps partly bleached olive-stones, or precisely similar tissue; black poivrette is the same, mixed with a little black husk. It is to be noted that, although it contains no starch, yet it yields some sugar to Fehling's solution, after being boiled for some time with dilute hydrochloric acid. The quantity depends on the length of time and strength of acid, but may be stated approximately about ten per cent. It is important to bear this fact in mind when making a full chemical analysis of pepper containing poivrette. After removing from such a mixture the matters soluble by boiling in dilute caustic alkali, the woody fibre which remains has a yellow colour; it consists of the poivrette, and some of the cells of pepper-husk and one of the subcortical layers of the pepper-berry. The pepper-cells are made lighter, and the poivrette-cells darker by the alkali, so that the two are more nearly of a similar yellow colour after treatment with alkali. This renders it more difficult to distinguish such of the cells as have somewhat similar markings; but it enables us to distinguish more clearly, as poivrette, the many torn particles which have no definite form or markings. The final examination of the complete cells is better made with good daylight rather than with artificial light, and in a portion which has been treated with water only.

The pepper cells are mostly different in shape, and are coloured, and have generally a dark substance in the interior. They are not numerous, but the quantity varies in commercial samples, owing to the modern practice of decorticating the pepper berry to every different extent possible, and mixing the various portions so obtained, including husks, in every variety of proportion with each other or with ordinary pepper. Each individual analyst must make himself familiar with both kinds of cells, as no description can convey an adequate idea of either. In order to form a judgment regarding the proportions of the different chemical constituents of commercial samples, we require to know the chemical composition of the different layers of the pepper-corn; and I hope soon to communicate to the society some figures bearing on this point, as well as to notice some other substances used in the sophistication of pepper.

It is interesting to note that the exemption, mentioned in section 8 of the Sale of Food and Drugs Act, in the case of a label being affixed to the article sold, intimating that the same is a mixture, does not apply in the case of poivrette, the admixture being made manifestly for the purpose of fraudulently increasing the weight and bulk.

Liverpool, 4th January, 1887.

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## ON THE ESTIMATION OF METHYLALCOHOL IN PRESENCE OF ETHYLALCOHOL.

BY OTTO HEHNER.

*Read at the Meeting, January 12th, 1887.*

THE readiest analytical method of which I know for determining the amount of methyl-alcohol, when mixed with ordinary alcohol is that of Dr. Dupré, described in the ANALYST



of 1876. He oxidises a known quantity of the alcohol with bichromate of potash, acidified with sulphuric acid, distils from an oil bath the acetic acid formed, and estimates by decinormal soda. Only ethylalcohol, according to Dr. Dupré, furnishes acetic acid, methylalcohol being completely oxidised into carbonic acid. Knowing the total alcoholic strength of the fluid to be examined—the specific gravities of methyl and ethylalcohol being nearly identical—the deficiency of acetic acid obtained indicates the proportion of methylalcohol in the mixture.

Dr. Dupré also takes the vapour tension of the alcoholic fluid in Geissler's vaporimeter, the readings on that instrument being higher, if methylalcohol is present, than corresponds to the specific gravity.

The only test experiment given by Dr. Dupré, in which 1 per cent. of wood-spirit (or rather 10 per cent. of methylated spirit) was added to a sample of whisky, indicated .42 per cent. of methylalcohol.

Having lately to examine a number of methylated spirits for the percentage of wood-spirit contained in them, I took occasion to investigate the subject, and in the following give my results.

First, as to the indication of the vaporimeter.

A sample of methylalcohol, purchased as pure, sp. gr. .8097, diluted to 10 times its bulk with water, gave a vaporimeter indication of 14.9, ethylalcohol of the same strength requiring 9.7.

Rectified wood-naphtha, sp. gr. .8107, similarly diluted, gave vaporimeter indication 12.0, against 9.6 of ethylalcohol.

Crude wood-naphtha, such as is used for methylating, sp. grav. .8233, gave an indication of 11.5, against 9.3 of ethylalcohol.

Ethylalcohol, .7969 sp. grav., showed 10.0 instead of 9.99.

Methylalcohol, purified with calcium chloride, boiling at 67 °C, sp. grav. .8129, vaporimeter indication 9.0, instead of 9.6.

This latter result, repeated many times, is not a little remarkable. The alcohol, as found by further investigation, was not quite pure, yet it consisted to a very large extent of methylalcohol, which, boiling at a low temperature, yet gave an indication *lower* than ethylalcohol of a corresponding strength would have done.

Looking at the wide variation of the differences between the vaporimeter indications and the corresponding ethyl indications, I have to come to the conclusion, that it is not the methylalcohol, but the impurities (acetone, etc.) contained in the methylalcohols, which are measured by Geissler's instrument, and that this may give, in case of impure methylated alcohol being employed, qualitative indications of the presence of methyl in ethylalcohol; yet it is quite useless as a quantitative instrument for this purpose.

Having some previous experience of Dr. Dupré's oxidation method, I had experienced the difficulty of distilling off in the oil-bath all acetic acid formed from the tenacious mixture of sulphuric acid and chromium and potassium sulphates. With patience and time, this can be done, but the operation is a troublesome and tedious one. I determined therefore to measure the quantity of chromate reduced, instead of the acetic acid produced, especially as I believed that much more decided indications could thus be obtained, than by a method, by which the most valuable constituent, the methylalcohol, was really not estimated at all.

Theoretically, one equivalent, or 46 parts, of ethylalcohol require two equivalents of oxygen for transformation into acetic acid, whilst one equivalent, or 32 parts, of methylalcohol, want three of oxygen for oxidation into carbonic acid. 1 mol. of bichromate (295.18) gives off three of oxygen. Hence

100 parts of ethylalcohol want 427.8 parts of bichromate, and

100    "    methylalcohol    "    222.4    "    "

I made solutions of bichromate, 80 grms., with 150 c.c. strong sulphuric acid to the litre, and of ferrous and ammonia sulphate, about 240 grms. to the litre. Also, by dilution, a chromate solution ten times more diluted than the one above mentioned. The chrome solution was accurately standardised upon the iron solution, the usual ferricyanide reaction being taken as indicator. 1 c.c. of iron solution was thus found to be equal to about .17 to .18 of strong chromate.

A quantity of alcohol, of known specific gravity, is diluted, at 60° F., to 100 c.c.; of this diluted alcohol a volume is taken, containing about .2 gram. of absolute alcohol. 25 to 30 c.c. of strong chromate are mixed with that quantity of alcohol in an ordinary 8 oz. bottle, securely corked, and the mixture heated in the water-bath for *at least* two hours. An excess of iron solution is then run in, and this titrated back with decichromate solution. I found that thus the end-point of the reaction could much more readily be observed by the gradual disappearance of the blue ferricyanide colour than by the direct titration with iron solution. The volume of iron solution is then calculated into the strong chromate which corresponds to it, and this subtracted from the total chromate added. The remainder, multiplied by .08, gives the weight of bichromate reduced.

Thus I diluted 5 c.c. of pure ethylalcohol of .8344 sp. gravity, corresponding to 85.5 per cent. by weight of absolute alcohol, to 100 c.c., and took 5 c.c., corresponding to .25 c.c. of the original alcohol. These .25 c.c., therefore, contain .1783 gram. ethylalcohol. These were oxidised, for two hours, with 25 c.c. of strong chromate. 85 c.c. iron solution (1 c.c. of which was equal to .188 c.c. chromate), were run in (equal to 15.98 chromate), and the excess of iron titrated back into 3.3 c.c. decichromate. Thence  $25.33 - 15.98 = 9.35$  c.c. of chromate, containing .7480 gram. of bichromate were reduced, corresponding to 419.6 per cent. of bichromate were consumed by the alcohol. Or 98.1 per cent. of the quantity of alcohol taken were found.

A sample of the purest methylalcohol which I could obtain, prepared from winter-green oil, and possessing a slight odour of the latter, of .8153 sp. gravity, and boiling at 66°C., heated for half an hour, consumed 829.2 per cent. of bichromate, corresponding to 81.2 per cent. of methylalcohol; in one hour 891.5 per cent., equal to 93.8 per cent. methylalcohol; and in two hours 908.8 per cent. of chromate, equal to 97.3 per cent. of pure methylalcohol, all results calculated upon the anhydrous alcohol, as taken from the ethylalcohol table.

Now, strictly speaking, the specific gravities of methyl and ethylalcohol are not alike. Allen calculates, from various sources, that at 15.5, pure methylalcohol has a density of .8021, corresponding to 97.33 per cent. of ethylalcohol of the same density, which is in absolute accordance with the above result.

In the case of mixtures of pure methyl and ethylalcohol, we would have to calculate as follows:—222.4 being the percentage of bichromate used by methyl, and 427.8 by



ethylalcohol, a difference of 494.6 beyond 427.8 would indicate 100 per cent. of pure methylalcohol. Hence subtract 427.8, the percentage consumed by any particular sample, and divide the remainder by 4.946, to obtain the percentage of methylalcohol in the sample, calculated upon the *absolute* alcohol. By a simple further calculation we would get percentages on the hydrated samples.

Take the case of the following mixtures, in which pure ethyl and pure methylalcohol, the former containing .03567 grm., the latter .03778 grm. in each c.c., were mixed. 1 c.c. dilute ethyl and 4 c.c. methylalcohol (the mixture containing 80.8 per cent. methylalcohol, calculated upon the absolute alcohol). Bichromate consumed 808.6 per cent., corresponding to 76.9 per cent. methylalcohol.

2 c.c. ethyl, and 3 c.c. methylalcohol (with 61.3 per cent. methylalcohol) used 720.3 per cent. bichromate, corresponding to 59.3 per cent. methylalcohol; 3 c.c. ethyl and 2 c.c. methylalcohol (with 41.4 per cent. methylalcohol), used 620.3 per cent. bichromate, equal to 38.9 per cent. methylalcohol. On making the allowance of 97.3 indicated methyl, being 100 per cent. real methylalcohol, and the figures stand as follows:—

Found		Taken
79.0	.. ..	80.8
60.9	.. ..	61.3
40.0	.. ..	41.4

These figures show that, for mixtures of the pure alcohols, the method gives results of sufficient practical accuracy.

Now as to methylated spirits.

Crude wood spirit, such as is used for methylating, required 810.9 per cent. of bichromate, and contained, therefore, 77.7 per cent. of methylalcohol.

Various methylated spirits, purchased at different shops, gave the following results:—

Bichromate used,				Corresponding to Methylalcohol
466.5	..	..	..	7.8
462.9	..	..	..	7.1
451.9	..	..	..	4.9
450.8	..	..	..	4.7
455.0	..	..	..	5.5
456.3	..	..	..	5.7
456.3	..	..	..	5.7

I have also met with two samples which were decidedly *over*-methylated; they used

554.3 bichromate equal to 25.5 methylalcohol.

546.4       "       "       23.9       "

These samples had been exported to Australia, where the excise authorities declared them to be insufficiently methylated. They were, therefore, re-methylated, at the sender's cost. The spirits had, according to the above figures, a considerable excess of methylalcohol.

Owing to the variable composition of crude wood-spirit—stated by Allen to contain from 35 to 95 per cent. of real methylalcohol—it is, of course, impossible to calculate the percentage of actual wood-spirit in any of the samples, unless the identical wood-spirit which has been used in mixing is also analysed. But the figures given furnish an idea of

the composition of methylated spirits, and may form a rough basis of calculation, in case of whiskies or other spirits, that may be found to consume an excessive quantity of bichromate. I have not met with any such, although I think an extensive search might furnish interesting results. On the contrary, my experience, as far as it goes, shows that whiskies and brandies, when examined by my method, consume a quantity of bichromate decidedly *short* of the theoretical amount.

A whisky, with .05 per cent. of total solids, used 405 per cent. of bichromate, equal to 95.2 per cent. of ethylalcohol in the absolute alcohol of the whisky.

Another sample (distilled) used 405 per cent. of bichromate, or 94.7 per cent. of ethylalcohol.

Another sample used but 399 per cent. of bichromate, corresponding to 93.4 per cent. of ethylalcohol.

A fourth sample required 401.1 per cent. = 93.8 per cent. of ethylalcohol, whilst in two other cases 401.1 and 399 per cent. of bichromate were reduced, indicating 93.8 and 93.4 per cent. of alcohol.

A sample of brandy used 406.2 of bichromate = 94.9 per cent. of alcohol.

In the whole of these cases the heating with bichromate was continued for so long a time that I have no doubt of the completeness of the oxidation. In proof of this I will quote duplicate estimates performed upon a sample of alcohol (probably, also, not absolutely pure) this reduced bichromate corresponding to 98.2 and 98.1 per cent. of ethylalcohol. Very many, indeed, of the figures which I have quoted in the paper were confirmed by duplicate experiments.

I do not as yet feel myself in a position to give anything like a definite opinion as to the cause of this deficiency. I cannot, however, help thinking that whisky and brandy are far from being fairly pure alcohols, but that they contain tangible percentages of substances of higher molecular weight. I will not fail to investigate this subject without delay, and to bring the results before the Society at an early date.

I beg to record my obligation to my assistant, Mr. Henry Richmond, who has assisted me in carrying out this investigation.

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#### ON SOUR BREAD AND THE LOGWOOD TEST.

By W. C. YOUNG, F.C.S., F.I.C.

*Read at the Meeting, December 8th, 1886.*

In applying the logwood test to bread it frequently happens that a reaction is obtained which further examination shows is not due to the presence of alum. This is more often observable when a pale tint is employed, and then the blue colour is seen on the margin of the stain, or near the outer parts of the loaf. In cases of this kind I have always felt, in giving a certificate of genuineness, that there was some doubt about it, and although I have repeatedly made special examinations of the sample for iron and other metals, I have never been able to account for the reaction.

Quite recently, circumstances occurred which enabled me to clear up the mystery,



and as the results of my experiments are of some interest, and perhaps importance, to Public Analysts, I have thought it advisable to lay them before the Society. On commenting, in a quarterly report to one of my boards, on some prosecutions for adulterating bread with alum, I was asked by one of the members present, who is a baker in a large way of business, whether it was possible for me to mistake sourness in bread for the presence of alum. Of course I replied without hesitation that it was quite impossible. He then said that he had a very good reason for putting the question, as some years ago, having had considerable trouble in making his bread, which frequently became sour, he sent a sample of the flour used and a sample of the bread made from it to a late eminent chemist, in the hope that analysis would afford him some explanation, and suggest some way out of the difficulty. The chemist reported that the flour was perfectly genuine, but the bread contained a large quantity of alum. As he (the member of the Board) was quite sure that no alum had been added to the bread, he concluded that the chemist had mistaken sourness for alum. Some time after this I received a sample of bread from this Board, which had been purchased and submitted for analysis by a private resident in the parish. This sample was heavy, dark in colour, and sour. Upon applying the logwood test in the usual way a very distinct reaction was obtained, and I felt convinced that it contained a large quantity of alum, but upon analysis I could obtain only aluminum phosphate equivalent to 7.8 grains of alum in 4 lbs., a second estimation giving me 8 grains of alum in 4 lbs. It was therefore perfectly clear that the reaction was not due to the presence of alum, and further examination failing to give me any indication of the cause, I certified the bread to be free from alum or other mineral adulterant, but felt strongly that my results were unsatisfactory.

Subsequently I learnt that this sample had been prepared by the before-mentioned gentleman expressly to test my ability to distinguish sour bread from bread containing alum.

In order to settle this question I made some bread sour by moistening it with water, and setting aside for a few days, and was surprised to find that in every instance I obtained the blue colour with the logwood test.

I got the same result with bread made from sour dough, although the bread contained alumina equal to only 6 grains of alum in 4 lbs.

As the acids produced by the fermentation of flour *per se* give no reaction with the logwood test, the effect must be caused by the solution of the alumina naturally present in the flour by the acetic acid formed, and this seems to be the true explanation.

I have moistened twenty different samples of bread, all of which had previously given negative results to the logwood test, with dilute acetic acid (about 1 of acid to 250 of water), and in one hour all of them gave a most intense blue colour. Flour made into a paste with weak acetic acid, and set aside for a short time has invariably given me the alum reaction. The depth of the colour obtained is such as we are accustomed to see when 50 or 60 grains of alum in 4 lbs. of bread are known to be present, and assuming that the whole of the alumina naturally existing in these samples was dissolved by the acetic acid, it is surprising that so minute a quantity should give so decided a reaction.

As a bread, to which say 10 grains of alum in 4 lbs. has been added in the making, gives a much less distinct blue colour than that to which none was added, but which has been moistened with acetic acid, it would seem to indicate that a large proportion of the alumina in the alum in the former case has been rendered insoluble. If this is so it is possible that the whole of the alumina in the alum added to bread may, in some cases, become insoluble, and then the logwood test would fail. This investigation throws grave doubt on the efficiency of the logwood test as an indicator of the presence or absence of alum in bread, as if a reaction be obtained it may be due to acidity, and not to alum, and if no reaction is given, then no proof is afforded that alum has not been added, as it may all have been rendered insoluble.

#### DISCUSSION.

The PRESIDENT said the paper was of great practical interest. They had hitherto been led to suppose that the blue colour was always a proof of the presence of alum, but it now appeared that was not so. However, it was an error in the right direction—it was better than looking for alum when it was known to be present, and not finding it. If phosphate of alumina in presence of acid did give the blue tint, then the assumption that the test only acted with alum could not apparently be borne out. He would, without himself pronouncing any opinion at the moment, be glad to hear the criticisms of members present on Mr. Young's remarks.

Mr. BODMER said that he had noticed certain breads free from alum, which gave a dirty slate-blue colour something like the alum tint. He would like to know if any member could account for this.

Dr. WINTER BLYTH said he always tested for alum by the gelatine process. By this method gelatine is soaked in the bread paste and then tested with logwood, when it gives a neater reaction. Mr. Young appeared to throw doubts on the applicability of the logwood test. According to his own showing, however, it was still a test for alumina, and his explanation was that the acetic acid acted in a solvent way upon the alumina, and so caused the colour.

So far as he (the speaker) knew, no one had been able to separate alum as alum in bread, and still the logwood test held good for alumina. All bread contained more or less alumina, and according to this explanation all bread would give the reaction if sufficiently sour, but bread was almost invariably acid, and if it were steeped in water the water would be found distinctly acid. It would be more satisfactory if the spectroscope had been used, because there was a difference in the spectroscopic appearance of the alkaline logwood with and without the real alum colour.

Mr. JOHNSTONE inquired what baking-powder had been used for the bread. He had had some powder containing 49 per cent. of alum, the rest being carbonate of soda. As to testing with the spectroscope, he thought it would be better to test quantitatively for the amount of alumina at once.

Mr. DYER said that acetic acid did not dissolve phosphate of alumina, which was insoluble, and it was rather a gratuitous assumption that the alumina put into bread was phosphate. It might be true that if they made a watery decoction of the bread they might make it into a phosphate, but it was not certain.

Dr. MURER said that lumps of actual alum could still occasionally be found in bread. He had obtained a piece about five years ago a little bigger than a pea. In reference to the slate colour mentioned by Mr. Bodmer, about fifteen years ago there was a great dispute in the West of England about some bread which was found to have been made with a special tartrated baking-powder, and which, on being tested, gave the slate colour, although free from alum. Therefore, accidental colours, due to other causes than alum,



were no new thing. The simple way was to estimate the alumina quantitatively, and not trust entirely to a colour reaction.

Mr. YOUNG, in reply, said that each bread had been previously tried with the log-wood test, and gave negative results before acidulating. He thought the question all turned upon whether the alumina was converted into phosphate of alumina or not; what he admitted to be quite possible was that the phosphate might be in combination with the gluten, and that that compound was soluble. It seemed to him that when they got no reaction with bread in the ordinary way, and then, after having added an acid to it, they did get a reaction, it was only fair to assume that the acid was the cause of the reaction. He had not seen the slate colour alluded to by Mr. Bodmer. He had not used any baking-powder in the bread, which he made from sour dough. As to the gelatine process, he had never tried it.

#### ARSENIC IN GLAUBER'S SALT: ITS TOXIC EFFECTS.

By SIR CHARLES A. CAMERON, Ex-PRESIDENT, R.C.S.I., V.P., INSTITUTE OF CHEMISTRY.

*Read at the Meeting, January 12th., 1887.*

IN July, 1884, a farmer, John Donnellan, residing near Castlereagh, County of Roscommon, administered a purgative draught, consisting of Glauber's salt and infusion of senna, to each of his herd of twenty cows. The animals were not sick, but it is usual in Ireland to give an occasional purgative dose to cows, especially after any important change in their diet. In the course of a few hours all of Mr. Donnellan's cows became very sick, and on the following day ten of them died. The viscera of one of the cows were forwarded to me for analysis, as were also specimens of the Glauber's salt and senna, which had been administered to the animals.

The stomachs were slightly inflamed; but their coats were not corroded. They and their contents were found to contain small quantities of arsenic.

The senna was found to be pure.

The Glauber's salt received for analysis amounted to about one pound weight, and chiefly consisted of large crystals. It was found to contain arsenious acid, in the ratio of 8.4 grains of  $As_2O_3$  per pound of the salt. The arsenic was equally distributed throughout the crystals in the form of sodium arsenite.

It is difficult to account for the presence of a sensible proportion of arsenic in Glauber's salt. In the preparation of anhydrous sodium sulphate or salt-cake, highly arsenical sulphuric acid might be employed, but its arsenic would be volatilized as  $As_2Cl_3$  at the high temperature of the salt-cake furnace. A salt-cake which was prepared on a small scale, sulphuric acid containing nearly one per cent. of arsenic being employed, proved to be perfectly free from arsenic. Probably, the Glauber's salt in question was prepared from impure sulphuric acid, employed in producing some such by-product as, for example, the sodium sulphate obtained in treating iodine ley. I made every effort to trace the origin of the Glauber's salt, but failed.

It appears that to each cow one pound of the salt had been given, and assuming that in each dose there was  $8\frac{1}{2}$  grains of arsenious acid, it seems strange that so small a quantity could prove fatal to a cow. The evidence in favour of the view that the



ickness of the cows was due to the poisonous salt is, however, very strong. It is certain that the arsenic was in the most favourable condition for rapid absorption into the circulation, and it is not improbable that this poison, as well as others, may act more powerfully, when administered with aperients so rapidly absorbed as Glauber's salt is.

### NOTES ON THICKENED OR BLOWN OIL.

BY WILLIAM FOX AND JAMES BAYNES.

*Read at the Meeting, December 8th, 1886.*

Blows or thickened oils are prepared from the various seed oils, by heating the oil in a suitable tank to 70° C., and then blowing air into them through a perforated pipe; the temperature of the oil at once commences to rise, and irritating fumes are given off. The temperature is not allowed to rise beyond 75° C., otherwise the oil becomes of a dark colour. Acetic and acrylic acids are present in the fumes. The blowing is continued from sixteen to thirty hours, or until the desired specific gravity is reached. The changes which take place in the oil during this process are most interesting; from their ordinary fluidity they become of a syrupy consistency; their specific gravity increases up to 1.290; their insoluble fatty acids decrease to as low as 84.12 per cent. The glycerin, determined by the permanganate process, appears to increase about one per cent., except thickened linseed oil, which gives an increase of 2½ per cent.; this increase in the glycerin may be due to some soluble oxidation products of the oil, which become reduced to oxalic acid by the permanganate.

No great quantity of free acid is present in these thickened oils; the amount calculated into terms of oleic acid, in a number of samples, varied from 2 to 5 per cent. On saponification with alcoholic potash the soap is very dark.

The following are mean results obtained from thickened linseed, rape, and cotton oils:

Linseed:—

	Per cent.
Insoluble fatty acids .. .. .	87.67
Glycerin .. .. .	12.85
Free fatty acids .. .. .	2.73

The insoluble fatty acids, titrated with standard potash, gave 258.4 as the equivalent weight of the acid.

Cotton:—

	Per cent.
Insoluble fatty acids .. .. .	85.50
Glycerin .. .. .	11.68
Free fatty acids .. .. .	5.35

The insoluble fatty acids, titrated with standard potash, gave 196.0 as the equivalent value of the acid. From the Ba salt, soluble in alcohol, we obtained an equivalent of 188.0.

Rape:—

	Per cent.
Insoluble fatty acids .. .. .	84.70
Glycerin .. .. .	11.32
Free fatty acids .. .. .	3.70

Titrated with standard potash the insoluble fatty acids gave an equivalent of 186.0.

The equivalent 186.0, if the insoluble fatty acids were normal, would give the formula  $C_{11}H_{22}O_2$ . From the results of a combustion analysis, using fused chromate of lead, a large excess of oxygen was found, so that it would appear that the insoluble fatty acids are oxy-acids of the acrylic series.

### EXPERIMENTS WITH THE LACTOCRITE.

By A. WYNTER-BLYTH.

*Read at Meeting, December 8th, 1886.*

THE following table gives some determinations of milk fat by the lactocrite, as compared with estimations of fat by weight according to Adams' method. The analyses have been made, under my superintendence, by Mr. J. K. Colwell, the laboratory assistant at the St. Marylebone Court House. The table shows the greatest difference between the two estimations to be .14, and the mean difference .05 per cent. of fat in 100 of milk. When it is considered that, with practice, as many as four batches of twelve tubes can be charged, rotated, and read within the hour, it is evident that fat-estimations by the lactocrite are more speedy and more accurate than any other volumetric process yet devised. It will therefore prove a valuable instrument for technical purposes. I have to thank Mr. Barham and Dr. Faber for facilities in the carrying out of these experiments.

#### DETERMINATIONS OF MILK FAT BY THE LACTOCRITE.

No.	Specific Gravity.	Total Solids.	Ash.	Fat.		Difference.
				Lactocrite.	Adams.	
1	1033.0	12.90	.80	3.40	3.44	-.04
2	1031.0	14.12	.80	4.55	4.69	-.14
3	1032.0	13.12	.77	3.60	3.57	+.03
4	1030.5	13.07	.74	3.90	3.99	-.09
5	1032.0	12.98	.76	3.80	3.76	+.04
6	1031.5	14.27	.76	4.90	4.84	+.06
7	1032.5	13.84	.78	4.20	4.26	-.06
8	1030.5	13.00	.76	3.70	3.69	+.01
9	1031.0	13.51	.80	4.05	4.09	-.04
10	1034.0	11.76	.76	2.10	2.07	+.03
11	1035.0	9.99	.86	.45	.502	-.05

*Conclusion of the Society's Proceedings.*

#### NOTICE TO OUR READERS.

OWING to pressure of Society matter several original articles and all our abstracts are held over for next issue.

## COMMUNICATIONS FROM THE LABORATORY OF THE SCHOOL OF MINES, LEOBEN.

BY ED. DONATH AND RUD. GELLER.\*

1. DETECTION AND ESTIMATION OF SMALL QUANTITIES OF CHROMIC OXIDE.—The qualitative separation of very small quantities of chromic oxide from much ferric oxide, alumina and manganous oxide presents many difficulties, and often fails, when the usual systematic course is pursued, viz., fusion of the oxides with potassic carbonate and potassic nitrate. The following process works much better:—The solution which may contain, besides the metals already mentioned, the salts of the alkaline earths, is poured into a boiling solution of sodic carbonate containing some potassic permanganate. After boiling for a few minutes longer, the excess of permanganate is reduced by addition of a few drops of alcohol and filtered. Ferric oxide, manganic peroxide, alumina, and the carbonates of the alkaline earths are precipitated, whilst the filtrate contains the chromium as sodic chromate. If present in not too small amount, the liquid is coloured more or less yellow. If colourless, the liquid is concentrated to a small bulk, and acidified with dilute sulphuric acid. A piece of starch which has been moistened with a solution of potassic iodide is now introduced, and if chromium is present, will turn violet. Or the liquid may be shaken with potassic iodide and carbon disulphide, when the latter will turn violet.

These reactions are more delicate than the test with hydrogen peroxide. For the quantitative estimation we proceed in similar manner. The solution is slowly added to a hot permanganate, and boiled for ten minutes. The filtrate is acidified with hydrochloric acid, boiled with alcohol, and finally precipitated in a porcelain dish with ammoniac sulphide. In a mixture of 50 c.c. solution of ferric chloride (1 c.c. containing 0.0714 ferric oxide), 50 c.c. solution of alum (1 c.c. containing 0.0068 alumina), and 1 c.c. chromic chloride, containing 0.0018 chromic oxide, the latter was successfully quantitatively estimated.

2. VALUATION OF RICH COPPER ORES.—The usual plan is to first precipitate the copper by metallic zinc, then to redissolve precipitate after washing in nitric acid, and, after addition of ammonia, to titrate with potassic cyanide. The preliminary precipitation with zinc is advisable, as copper is but imperfectly separated from iron by ammonia. The following process will, however, be found more expeditious:—

The finely powdered sample is intimately mixed in a porcelain crucible, with double volume of zinc dust. The lid is put on, and the whole ignited for ten minutes. The ore is by this treatment completely desulphurised, the zinc taking up all the sulphur. After cooling, the mass is boiled in a beaker, with dilute sulphuric acid (1-4), which dissolves the excess of zinc and the zinc sulphide, also the iron. The copper is left insoluble, but contains, besides small quantities of cupric sulphide, any metals precipitable by zinc which may have been present in the ore, also the gangue. After thorough washing with boiling water, the mass is, as usual, treated with nitric acid, filtered, diluted up to a definite bulk, and aliquot parts are titrated with potassic cyanide.

\* Repert. Anal. Chemie, 3, 1887.



3. CHANCEL'S PROCESS FOR THE SEPARATION OF ALUMINA FROM IRON.—The direct separation of alumina from iron is best effected by boiling the neutralised solution with sodic hyposulphite (Chancel). In practice, the process is but seldom employed, most chemists preferring to weigh the mixed oxides, and then to volumetrically estimate the iron. We have, however, made some experiments to test the accuracy of the method.

A very weak solution of alum, 30 c.c. of which yielded .0768 alumina, was mixed with 25 c.c. of a neutral solution of ferric chloride (= .1785 ferric oxide) diluted with water up to 400 c.c., and boiled down to about 100 c.c., after addition of excess of sodic hyposulphite. The precipitated alumina was then at once filtered off. The following results were obtained:—

Number of c.c. of solution of alum taken.			Containing Alumina			Alumina Found
30	..	..	.0764	..	..	.0730
—	..	..	—	..	..	.0730
40	..	..	.1018	..	..	.0984
—	..	..	—	..	..	.0956
50	..	..	.1273	..	..	.1220

As will be noticed, the precipitation of the alumina was in no case complete. The filtrate and washings, of the two last experiments were boiled down to half the bulk, and further precipitates of alumina were obtained, amounting to .0072 and .0068 respectively. This, however, makes the results too high, which is explained by the difficulty of completely expelling the co-precipitated sulphur. The presence of sulphur may be detected by the yellowish colour the mass assumes on ignition. Alumina, free from sulphur, does not turn yellow. Chancel's process is, however, trustworthy, when the following rules are adhered to: strong dilution, and boiling down to at least half the volume, again boiling the filtrate, and collecting any further precipitate of alumina, and igniting precipitate before the blowpipe until yellow colour disappears.

4. ON THE USE OF HYDRIC PEROXIDE.—Hydric peroxide is frequently used in analytical work, for instance, to completely precipitate manganese from its ammoniacal solution. We have conducted some experiments, to see whether other metals would interfere with the accuracy of the process.

Fifty c.c. of a solution of manganous sulphate, containing .1335 manganous manganic oxide, were mixed with a weak solution of zinc sulphate, mixed with excess of ammonia, and boiled with hydric peroxide. The precipitate was rapidly washed, and ignited before the blowpipe. Two other experiments were made, using nickelous sulphate, and calcic chloride. The following results were obtained:—The first precipitate weighed .1540 gramme, the second .1475 gramme, the third .1376 gramme. As these results were too high, the precipitates were redissolved in hydrochloric acid, and again precipitated with ammonia and hydric peroxide, when the following weights were obtained: .1334, .1467, .1341 gramme. From these experiments we may conclude the following:—Manganese may be completely separated from zinc by repeated treatment (about four times), with ammonia and hydric peroxide. Two precipitations will free it from nickel, but from lime the separation is incomplete.

Precipitated cobaltous oxide is quickly oxidised to the sesquioxide by ammoniacal hydric peroxide, but nickelous oxide does not alter. Ammoniacal solution of nickel does

not change on boiling with the reagent, but ammoniacal solution of cobalt colours deep brownish-red. On acidifying with hydrochloric acid and boiling, nearly all the cobalt precipitates as the roseo compound. This reaction is very useful for preparing pure cobalt compounds from cobalt ores containing nickel.

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REVIEWS.

SANITARY EXAMINATIONS OF WATER, AIR AND FOOD: A *vade mecum* for the Medical Officer of Health. By Cornelius Fox, M.D. Second Edition. London: J. and A. Churchill, New Burlington Street, W.

THIS concise, but yet distinct, treatise has undergone considerable change and vast improvement since the first issue. It has the great advantage of having a real *raison d'être*, because it is written on definite lines, and with a fixed purpose. The author's views on the relative duties of the medical officer and of the public analyst are sound, and ought to be more generally appreciated by health authorities. He holds that, between the duties of the two officers, there is a strict and well defined line. The medical officer should be competent and trained to undertake the examination of water, air, and food from a *strictly sanitary* point of view; he should be able to make such chemical and microscopical analyses as will enable him to say whether the water and air of any district are or are not in a state conducive to a healthy condition of the inhabitants. In the domain of food his duties should be limited to searching for any condition dangerous to public health, such as decayed or diseased meat, fish, milk, bad flour, and decomposed or poisonous food generally. There the functions of the medical officer should cease and those of the public analyst commence. It is the duty of the latter officer to examine all food specially for what are strictly adulterations and frauds with the commercial object of extra profit. In a word, the medical officer is to look after the *health* of the people, while the public analyst is the lever whereby the local authorities can prevent their constituents from being swindled in pocket. This being the author's opinion, he writes accordingly, and with very excellent effect. The information given is of the most recent nature, and in water analysis the bacteriological mode of investigation is fully described. The whole book is well put together, and will form an excellent help to those medical men presenting themselves at the special examinations in sanitary science recently established at the various universities. The illustrations of water deposits and of parasites in meat, etc., are distinct and well drawn, and the print is of that large, plain style, so delightful to the tired eye of the student. Dr. Fox's work has made its mark as a real text-book, likely to now run through many editions.

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OUTLINES OF QUANTITATIVE ANALYSIS. By A. Humboldt Sexton, Professor of Chemistry in the College of Science and Arts, Glasgow. London: Charles Griffin and Co., Exeter Street, Strand.

THE author's excuse for adding yet one more stone to the pile of works on chemistry is that he has found the want of a book for use in the laboratory sufficiently short and simple for ready reference by students who desire a fair knowledge of the ordinary quantitative work of a chemist, and yet sufficiently comprehensive for all common cases. The plan is something like that of a very reduced "Fresenius," but only one method for each case is definitely chosen and adhered to. It must be admitted that the processes so selected are, in most cases, those in actual common use in our commercial laboratories, and that the choice generally has been judicious. It is certain that, given



proper laboratory supervision and oral instruction, the student working by a book like this will become a fairly expert mineral analyst with much less trouble than before. His ideas will, of course, be somewhat dogmatic and narrow, but he will have a really good foundation to start with, and then he can amplify his methods from larger works at his leisure. After taking the estimation of the common metals and acids, both gravimetrically and volumetrically, we have the separation of the chief metals and acids, and the book winds up with a special chapter devoted to the analysis of no less than twenty-three of the ordinary minerals and commercial products commonly analysed for trade purposes. We could point out one or two little matters wherein the author could improve the next edition, but we do not want to be hypercritical over a book written with excellent intention, and likely to be exceedingly useful to a large number of students of quantitative analysis.

### CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

To the Editor of the ANALYST.

SIR,—I regret that my absence in Denmark prevented me from being present at the meeting of the S.P.A. on December 8th, when my paper on "The Lactocrite" was read, and I shall be obliged if you will allow me space for a few lines in reply to some remarks made after the reading of the paper.

Mr. Adams was not quite correct in seeing in my paper (at least, not directly) a corroboration by the lactocrite to his own method of analysing, because I have used Storch's method for the analyses quoted, which method has also been used by Mr. Sebelien. The analyses in my paper show a close correspondence between the lactocrite and Storch's method. Mr. Adams' remark may perhaps have reference to the equally good results obtained by Dr. A. Wynter Elyth in trying the lactocrite, as he used Adams' method. But, if both Storch's and Adams' methods correspond with the lactocrite, how can they fail to correspond between themselves, as Mr. Adams says they do? Fortunately he gives himself the explanation, as, from his description of Storch's method, it appears that he has overlooked the most important feature. It is not claimed that pumice-stone, when used simply as an absorbent, is in any way superior to sand, plaster of Paris, or similar media. The advantages found in pumice are that it is brittle and highly spongy, and that it therefore easily can be reduced to a very fine powder. In this state only should it be extracted, viz.: the milk is thoroughly dried on the pumice gravel, and this is then ground very fine—almost as flour. It is claimed that no fat can escape extraction when this fine powder of milk and pumice is repeatedly treated in an extraction tube of the description correctly given by Mr. Adams. Mr. Adams has—if I understand his remarks correctly—extracted the coarse pumice powder (gravel) without pulverising it, and this is the only explanation of the fact that he has found a difference between his own very able method and that of Storch.

Dr. Vieth's remark on the testing of skim-milk with the lactocrite I hope to be able to answer later on, when I shall give the results of some experiments with the lactocrite as applied to separated milk.

—I am, sir, yours truly,

HARALD FABER.

London, January 12th, 1887.

### BOOKS, &c., RECEIVED.

AMERICAN Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; British and Colonial Druggist; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Hospital Gazette; Illustrated Science Monthly; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Scientific American; Society of Arts Journal.

### NOTICES TO CORRESPONDENTS.

ALL Communications to be addressed to 325, Kennington Road, London, S.E.



# THE ANALYST.

MARCH, 1887.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of the Society was held at Burlington House on Wednesday, the 9th of February, the President, Mr. A. H. Allen, in the chair.

The minutes of the previous annual meeting were read and confirmed.

On the ballot papers being opened, the following gentlemen were found to be elected:—As member, Herbert L. Buckeridge, F.C.S., analytical chemist, Crouch End. As associate, F. P. Seddall, assistant to Mr. Young.

The following gentlemen were proposed for election, and will be balloted for at the next meeting:—As members, Dr. Theophilus Redwood, F.C.S., F.I.C., public analyst for Middlesex; T. Horne Redwood, London; A. Norman Tate, Liverpool; J. E. Stead, public analyst for Middlesbrough. As associate, H. Richmond, assistant to Mr. Hehner.

The following papers were read and discussed:—

On the Composition of Milk and Milk Products, and

Notes on the Complete Analysis of Milk, by Dr. P. Vieth, F.C.S.

Notes on the Estimation of Glycerine in Fats, by Otto Hehner.

A convenient apparatus for the estimation of sugar was exhibited and explained by Mr. A. Stokes.

A further communication from Dr. Campbell Brown, "On Poivrette, the New Pepper Adulterant," was read by Dr. Muter.

The next meeting of the Society will be held at Burlington House on Wednesday, the 9th inst.

## ON THE COMPOSITION OF MILK AND MILK PRODUCTS.

BY DR. P. VIETH, F.C.S.

*Read at the Meeting, Feb. 9th, 1887.*

THIS is the sixth time that I bring before you what may be called an annual report on the work done in the laboratory which is under my charge. If the yearly repetition of statements of a very similar character may appear to some unnecessary and tedious, others, I hope, will find them increasing in value with the extension of the period which they cover. In order to avoid going again over the same ground, I shall not speak on the circumstances under, and the purpose for, which the samples were taken, but refer you, with regard to these points, to former communications. (THE ANALYST, VII. p. 53, VIII. p. 33, IX. p. 56, X. p. 67, and XI. p. 66.)

The total number of analyses made during the year 1886 is 18,862, of which

17,269 are analyses of milk,

1,082	"	"	"	cream,
355	"	"	"	skim milk,
34	"	"	"	buttermilk,
27	"	"	"	butter and butter fat,
10	"	"	"	cheese,
48	"	"	"	milk preparations for infants and invalids,
13	"	"	"	water,
24	"	"	"	sundries.

Of the milk samples 12,181 were taken from the railway churns on their arrival in the dairy, while 4,135 were taken by the inspectors employed by the business, from the men, when delivering the milk to the customers. Of all these samples specific gravity and total solids were determined, and the percentage of fat calculated from these two items, using Fleischmann's formula (*THE ANALYST*, XI. p. 68). The monthly averages of the results are contained in the following table:—

AVERAGE COMPOSITION OF MILK.

1886.	SAMPLES TAKEN.				
	ON ARRIVAL.				ON DELIVERY.
	Spec. Grav.	Total Solids.	Fat.	Sol. n. f.	Total Solids.
January .. ..	1.0322	12.87	3.77	9.10	12.78
February .. ..	1.0322	12.83	3.73	9.10	12.82
March .. ..	1.0323	12.78	3.69	9.09	12.74
April .. ..	1.0321	12.75	3.70	9.05	12.71
May .. ..	1.0323	12.80	3.71	9.09	12.79
June .. ..	1.0322	12.78	3.70	9.08	12.74
July .. ..	1.0318	12.67	3.69	8.98	12.67
August .. ..	1.0319	12.77	3.74	9.03	12.77
September .. ..	1.0321	12.98	3.89	9.09	12.93
October .. ..	1.0321	13.26	4.11	9.15	13.09
November .. ..	1.0322	13.32	4.14	9.18	13.15
December .. ..	1.0324	13.27	4.06	9.21	13.13
Yearly Average ..	1.0322	12.92	3.83	9.09	12.86

Considering the circumstances under which the samples were taken, the differences between the two series must be looked at as very insignificant.

In all the samples of milk, which were not mere control samples, the fat was determined gravimetrically, employing what is known as Soxhlet's, or the plaster of Paris, process; in all there were 550 fat extractions made in this manner, which, however, do not relate to milk exclusively, but also to cream, skim milk, etc.

In addition to the two instances, which I have brought under your notice last year, with regard to the composition of ice formed in milk (*THE ANALYST*, XI. p. 68), I am in the position to-day to give you two more illustrations of what takes place when milk freezes. Milk, which arrived in a partially frozen state, was strained, the ice melted, the quantities of liquid and frozen part ascertained, and samples of each analysed with the following results:—



## COMPOSITION OF PARTIALLY FROZEN MILK.

	I.		II.	
	Liquid part.	Melted ice.	Liquid part.	Melted ice.
Water.. ..	87.10	91.83	87.21	92.46
Fat .. ..	3.87	2.56	3.57	2.16
Proteids* ..	3.21	2.28	3.50	1.96
Sugar .. ..	5.08	2.89	4.98	2.72
Ash .. ..	.74	.44	.74	.40
Spec. Gravity ..	1.0330	1.0190	1.0330	1.0180
Quantity of Ice ..	—	2 per cent.		2½ per cent.

Samples of cream, as supplied to customers, were taken regularly twice a day—morning and afternoon delivery—and further samples were taken by the inspectors from the men on the rounds; of the former 448, and of the latter 349, were submitted to analysis. The analysis is confined to determining the total solids, from which the fat is then calculated by means of a table, which I have drawn up and published three years ago (THE ANALYST IX. p. 61). The results of the examination were as follows:—

## AVERAGE AMOUNT OF FAT IN CREAM.

1886.	SAMPLES TAKEN.	
	Before sent out.	On delivery.
January .. ..	42.1 per cent.	43.2 per cent.
February .. ..	43.5 " "	43.5 " "
March .. ..	45.4 " "	45.7 " "
April .. ..	45.7 " "	45.6 " "
May .. ..	45.0 " "	45.7 " "
June .. ..	42.6 " "	42.0 " "
July .. ..	41.7 " "	41.5 " "
August .. ..	43.3 " "	43.5 " "
September .. ..	45.1 " "	45.8 " "
October .. ..	46.0 " "	45.2 " "
November .. ..	45.3 " "	44.7 " "
December .. ..	44.2 " "	44.4 " "
Yearly Average .. ..	44.2 per cent.	44.3 per cent.

The rather large differences, which occur between the two series of samples, must be assigned to various causes: firstly, absolute correctness cannot be expected from the mode of calculating the fat; secondly, the consistency of the cream makes thorough mixing and drawing real average samples rather difficult; and thirdly, the two kinds of samples do not exactly correspond with each other, the samples taken before sent out referring to morning and afternoon cream, while the samples taken on delivery refer

\* By difference.



to afternoon cream only. The morning cream being handed over to the men, and by them to the customers in sealed cans, taking control samples is not only superfluous, but impossible.

Calculating the fat in cream from the total solids, in the manner mentioned, has in my hands always given very satisfactory results, agreeing with the results arrived at when extracting and weighing the fat in far the larger number of cases, within .5 per cent. An addition of milk or water to cream, which does not alter the normal relation between fat and solids not fat, does not interfere with the calculation. In cases, however, where the relation between fat and non-fatty solids has been disturbed, the table for calculating the fat is not applicable; this is the case, for instance, with clotted cream. Of this preparation twenty-one samples were submitted to analysis, and found to be of the following composition:—

## COMPOSITION OF CLOTTED CREAM.

	Limits.	Average.
Water .. .. .	32.59 to 42.13	36.11
Fat .. .. .	50.36 to 61.43	57.36
Proteids and Sugar .. .. .	4.61 „ 8.93	6.01
Ash .. .. .	.42 „ .77	.52

For the determination of water, fat, and solids not fat, in products containing a very high percentage of fat, such as clotted cream, cream cheese, and butter, I am in the habit of using a method, which is virtually the same as the flask method proposed by Mr. Carter Bell, of Manchester, for the analysis of milk. About 5 grms. of the material are introduced into a conical flask, and dried to constant weight in an air bath at 100° C., the fat is thoroughly washed out with ether—an easy task, where so much fat is present—and the solids not fat, dried again and weighed. For the determination of the ash, a separate sample is taken, dried, and ignited.

In 74 samples of separated skim-milk, the fat was determined and found to amount to from .14 to .42 per cent. in 71 samples, while in 3 samples there were .55, .62, and .63 per cent. of fat present respectively.

The composition of 19 samples of butter varied between the following limits:—

Fat .. .. .	82.33 to 88.95	average 85.37
Water .. .. .	10.82 „ 15.94	„ 12.92
Proteids, etc. .. .. .	.08 „ 3.20	„ .97
Ash .. .. .	.02 „ 2.73	„ .74

Insoluble fatty Acids .. .. .	87.60 to 89.30	„ 88.32
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When the clarified fats were treated according to Reichert's method from 12.2 to 15.0, and in average, 14.0 cc. decinormal potash were required to neutralise the distillate.

In butter-fat about one year old and bleached by the action of air and light, the insoluble fatty acids were reduced to 84.03 per cent., and 15 cc. decinormal potash were required to neutralize the distillate from 2.5 grms.

Some time ago I have made you acquainted with the composition of an English cream cheese, containing an exceedingly high percentage of fat. (THE ANALYST XI. p. 163). Perhaps it might interest you, if I give you the analysis of another cream cheese made according to a French recipe:

## COMPOSITION OF FROMAGE GERVAIS.

	I.	II.	III.	IV.
Water .. .. .	47.94	45.69	42.07	33.57
Fat .. .. .	43.76	45.96	48.41	58.58
Casein, etc. .. .. .	7.49	7.60	8.77	7.14
Lactic Acid .. .. .	.29	.31	.22	.25
Ash, soluble .. .. .	.06	.06	.09	.04
„ insoluble .. .. .	.46	.38	.44	.42

The differences in composition are chiefly due to the different ages of the cheese, No. I. and II. being quite fresh, while No. III. was one, and No. IV. two days old when analysed.

The average composition of koumiss and other milk preparations for infants and invalids, calculated from analyses made in 1886 and preceding years, is given below.

## COMPOSITION OF MILK PREPARATIONS FOR INFANTS AND INVALIDS.

Water.	Alcohol	Fat.	Caseine	Albumen.	Lactoproteine & Peptones.	Lactic Acid.	Sugar.	Ash.		Remarks.
								Soluble	Insolu- ble.	
FULL KOUMISS.										
88.90	.15	1.35	2.01	.30	.34	.34	6.03	.17	.41	1 day old.
90.35	.94	1.36	1.96	.23	.53	.96	3.10	.23	.34	8 days old.
90.57	1.04	1.38	1.88	.20	.77	1.40	2.18	.23	.35	22 days old.
MEDIUM KOUMISS.										
87.55	.29	1.54	1.46	.43	.48	.68	6.80	.28	.49	1 day old.
88.39	.97	1.56	1.40	.25	.76	1.20	4.70	.32	.45	8 days old.
88.62	1.05	1.58	1.30	.14	.97	1.67	3.90	.33	.44	22 days old.
WHEY KOUMISS.										
89.74	.30	.11	.15	.39	.44	.60	7.48	.37	.42	1 day old.
90.63	1.03	.13	.14	.36	.49	.91	5.52	.37	.42	8 days old.
91.07	1.38	.15	.11	.32	.58	1.26	4.34	.37	.42	22 days old.
DIABETIC KOUMISS.										
92.24	.28	.51	2.19	.30	.36	.75	2.78	.22	.37	1 day old.
92.38	.35	.52	2.13	.25	.48	.86	2.42	.24	.37	8 days old.
92.55	.57	.51	2.05	.18	.65	1.22	1.64	.26	.37	22 days old.
RUSSIAN KOUMISS.										
91.87	.22	.34	2.32	.08	.32	.06	3.95	.46	.38	1 day old.
92.26	.45	.33	2.17	.07	.48	.31	3.08	.49	.36	8 days old.
92.52	.57	.33	2.03	.07	.63	.56	2.45	.49	.35	22 days old.
ARTIFICIAL HUMAN MILK.										
87.38	—	4.46	2.57			—	5.02	.22	.35	
SPECIAL MILK FOOD FOR INFANTS.										
89.28	—	3.77	1.49			—	4.95	.26	.25	
PEPTONIZED MILK.										
89.20	—	3.41	.96	.07	1.88	—	3.80	.25	.43	
WHEY.										
93.00	—	.09	.92			—	5.45	.31	.23	
BUTTER-MILK.										
90.19	—	.50	3.60			.80	4.06	.26	.59	
SKIM MILK.										
90.68	—	.29	3.49			—	4.76	.25	.53	

N.B.—In the analyses of the various kinds of koumiss, the carbonic acid present has not been taken into account.



With regard to samples which come under the head of "Sundries," I wish only to mention that I have examined a number of the powders which are so widely advertised and recommended as milk preservatives, and invariably found them to contain boracic acid. A liquid called "Anti-Bacterion," and recommended for cleansing purposes, consisted of a twenty per cent. solution of impure aluminium sulphate.

### ON THE ESTIMATION OF GLYCERINE IN FATS.

By OTTO HEHNER.

*Read at the Meeting, February 9th, 1887.*

IN the publication of the principle upon which the method to be described in the following paper is based, I have quite recently been forestalled by L. Legler (*ANALYST*, January 1887), and I, therefore, cannot lay any claim to originality. But as I operate in a manner quite different from that adopted by Legler—his process being only applicable to somewhat concentrated glycerine liquor, whilst I am enabled to determine the glycerine in even the most dilute solutions—I venture to lay a description of it, and of results obtained, before the members of the Society. A portion of my investigation is, furthermore, of general importance, and concerns all methods of glycerine estimation, since it treats of the question of the volatility of glycerine with aqueous vapour.

Glycerine decomposes, on treatment with bichromate of potassium and sulphuric acid, into carbonic acid and water. Legler weighs the carbonic acid, or rather the loss of carbonic acid, in an ordinary carbonic acid apparatus. Messrs. Cross and Bevan (*Chemical News*, Vol. 56, p. 2) measure the volume of the gas evolved.

It is evident that both these modifications require limited bulks of fluid, and, therefore, exclude the estimation of glycerine in very dilute solutions, such as are obtained in the analysis of fat—the washings, in fact, of the insoluble fatty acids.

The process which I have described (*ANALYST*, XII, February) for the estimation of methyl in the presence of ethylalcohol, and which consists in the measurement of the quantity of bichromate reduced, is, as I hope to show, particularly suitable for the analysis of such washings.

One part of glycerine requires, for complete oxydation, 7·486 parts of potassium bichromate.

*Solutions required.*—(1.) Bichromate, containing in each litre about 80 grammes of bichromate and 150 c.c. of strong sulphuric acid. The exact value of the solution should be ascertained by titration with solutions of known weights of iron wire.

2. Ferrous and ammonia sulphate containing about 120 grms. per litre.

3. Bichromate ten times more dilute than the above.

The ferrous solution is exactly standardised upon the chromate solution, and the glycerine value of the chromate (contents of  $K_2C_2O_7$  divided by 7·486) is calculated.

The chromate solution used in my experiments standardised as follows:—

2·8412 grms. iron-wire = 2·8327 pure iron, required 33·94 c.c. bichrome. 1 c.c. = ·07333 bichrome, or ·009796 glycerine.

2·7078 grms. iron-wire = 2·6997 iron used 32·3 c.c., bichrome 1 c.c. = ·07344



bichrome or .009810 glycerine. Average 1 c.c. bichrome = .07338 grms. bichrome or .009803 glycerine.

*Test Experiments.*—Glycerine, specific gravity 1.2572, containing, according to Lenz's tables, 95.55 per cent. of pure anhydrous glycerine—was taken, a solution of 12.5798 grms. per litre, corresponding to 12.0200 grms. of pure glycerine, being used. 25 c.c. of this solution, equal to .3005 glycerine were taken in each of the following experiments.

25 c.c. heated with 40 c.c. bichromate, without further dilution for two hours to near the boiling point: 30.41 c.c. bichromate were consumed, corresponding to .2981 grms. glycerine, or 99.2 per cent. of the glycerine taken.

25 c.c., heated with bichrome and 25 c.c. strong hydrochloric acid for one hour, consumed 31.16 c.c. bichrome, equal to .30546 grms. or 101.6 per cent.

25 c.c., diluted with 500 c.c. of distilled water, reduced in three hours 23.93 c.c. bichromate, equal to .23458 gm. or 78.1 per cent of glycerine.

The same quantity, diluted with 500 c.c. of water, plus 25 c.c. strong hydrochloric acid, heated for one hour, reduced 22.3 c.c. bichrome, indicating .2186 grms., or 72.7 per cent.

25 c.c., diluted with 500 c.c. of water, plus 25 c.c. sulphuric acid, consumed in one hour 29.34 c.c. bichrome, = .2876 grms., or 95.7 per cent. of the glycerine taken.

Conditions the same as in the previous experiment, only the heating continued for two hours. Bichromate consumed 29.89 c.c. = .2930 grms., or 97.5 per cent. of glycerine taken.

25 c.c. were diluted to about 300 c.c., the solution evaporated to about one-half upon the water bath, and then heated with 25 c.c. strong sulphuric acid and chromate. After two hours 30.54 c.c. bichromate were found to be reduced, corresponding to .2994 grms. or 99.6 per cent. of glycerine.

In a similar experiment the diluted solution was vigorously boiled down over the naked flame to about one half (the basin being, of course, covered with a dock glass to prevent loss by spurling), and heated for two hours with sulphuric acid and bichromate. Found .2961 grms. or 98.5 per cent. of the glycerine taken.

In a precisely analogous experiment 10 c.c. of strong alcohol were added to the water, before boiling, over the naked flame. After two hours 30.33 c.c. of chromate were reduced, equal to .2973 grms. or 98.9 per cent. of glycerine found.

The alcohol experiment repeated, but the fluid concentrated on the water bath, an amount of bichromate was reduced corresponding to 107.6 per cent. of glycerine. An odour of aldehyde was perceptible during the oxidation.

*Deductions.*—From these test experiments the following conclusions can be drawn.

1. In a fairly concentrated solution glycerine is quantitatively oxidised by acid bichromate.

2. In a very dilute solution the oxidation is not complete even after many hours' heating.

3. The addition of hydrochloric acid does not materially help oxidation.

4. In solutions containing about 10 per cent. of strong sulphuric acid the oxidation is complete after two hours, even in exceedingly dilute solutions (.6 glycerine per 1000 of fluid).

5. From such dilute solutions, glycerine does not, as is commonly assumed, volatilise on concentrating the fluid, be it on the water bath or over the naked flame.

6. Should alcohol be present it is completely volatilised by vigorously boiling the fluid down to one half, but not on the water bath.

The non-volatility of glycerine from dilute solutions may further be readily demonstrated by distilling from a large retort 500 c.c. of water, containing about .3 grms. of glycerine, catching the first 250 c.c. of the distillate. This distillate does not, even on heating, decolourise more than a few drops of a dilute permanganate solution, such as is used in water analysis.

*Method for Estimating Glycerine in Fats.*—Saponify about 3 grms. of the fat with alcoholic potash; do not drive off all the alcohol, lest glycerine should volatilise from the concentrated solution, but dilute to about 200 c.c.; decompose the soap with dilute sulphuric acid, filter off, and estimate insoluble fatty acids as usual. Vigorously boil the filtrate and washings (amounting to about 500 c.c.) in a covered beaker or basin, down to one-half, add 25 c.c. strong sulphuric acid (suitably diluted) and 50 c.c. standard bichromate. Heat to near boiling for two hours, and titrate back the excess of bichromate with excess of ferrous sulphate, and ultimately the latter with decichromate using ferricyanide as indicator. Calculate from the chromate consumed the amount and percentage of glycerine.

Finally, I will add a few results obtained on applying the method to a few fats:

Olive oil	..	..	..	..	10.26 per cent. glycerine.
Cod liver oil	..	..	..	..	9.87 per cent.
Linseed oil	...	..	..	..	10.24 and 10.20 per cent.
Butterine	..	..	..	..	10.01 per cent.
Butter	..	..	..	..	12.40 and 11.96 per cent.

Of course, I am fully aware that other substances, should they be present, might reduce bichromate as well as the glycerine does. The analogous objection applies to Dr. Muter's and Mr. Fox's processes; I have, however, convinced myself that soluble fatty acids, like butyric, do not act upon bichromate, nor do fatty acids of higher molecular value.

The method may not be theoretically perfect, but it may commend itself for its simplicity and rapidity in cases of fluids which cannot contain anything but glycerine and soluble fatty acids.

NOTE.—I here correct two errors that have crept into my paper on methylalcohol.

Dr. Dupré did find .58, not .42 per cent. of methylalcohol, for 10 per cent. of methylated spirit.

The ferrous sulphate solution employed contains 120, not 240, grammes of iron and ammonia sulphate per litre.

## A CONVENIENT APPARATUS FOR SUGAR ESTIMATION.

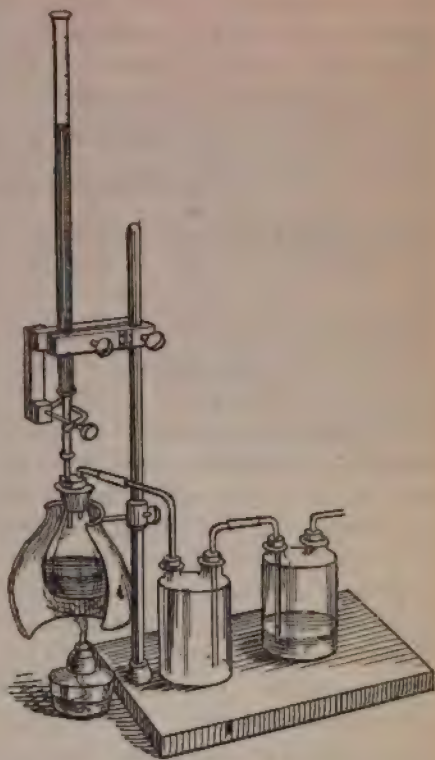
BY ALFRED W. STOKES, F.C.S.

In the discussion that ensued on the reading of Dr. Vieth's paper, Mr. A. W. Stokes, F.C.S., stated that the method he had used to verify the readings of Dr. Vieth's polariscope, was that described by Mr. Bodmer and himself in *THE ANALYST* (vol. X. p. 62). Dr. Pavy's ammoniacal cupric solution was used. A greatly improved apparatus was, however, employed; this he exhibited to the Society. We give herewith a drawing of



it,\* noting that it may be useful for other colour reactions. It consists of a burette one, lateral, half of which is of opal glass to show more sharply the divisions, and to mark accurately the centre of the meniscus. From this is pendent, by a short piece of india-rubber tubing, a flask of about 150 c.c. capacity. The cork used is of india rubber, with two perforations; into one of these fits a short glass tube, connected with the burette by the piece of tubing mentioned above, and dipping a short way into the flask, where it is drawn out to a moderately fine point. The other perforation is fitted with a tube leading off to two Woulffe's bottles, one of which is about one-third filled with water.

The tube between the flask and burette is best compressed by the specially-devised screw-tap figured. The ordinary spring-clip brings the fingers too close to the flame to be pleasant; nor does it allow so regular a succession of drops to fall. Behind the flask, to form a white background, is arranged the half of one of the ordinary white, opal gas-globes. By this white light is concentrated on the flask, and an admirable background formed. The method of using is described in the paper referred to above. The results obtained agree very closely with those of the polariscope.



#### FURTHER NOTE ON POIVRETTE.

By PROFESSOR J. CAMPRELL BROWN, D.Sc.

*Read at the Meeting, February 9th, 1887.*

IN addition to the microscope examination with a  $\frac{1}{8}$  objective in good daylight, a lower power, such as a  $\frac{2}{8}$  objective, with the polariser and analyser placed parallel, and with Klein's quartz plate or selenite plate, in daylight or strong lamplight, brings the ligneous cells into marked prominence, and enables the counting to be done on a tolerably large field. This does not distinguish between the cortical cells of pepper and poivrette cells. If, however, the quartz or selenite plate be removed, and the polarising and analysing Nicol prisms crossed, any power from a  $\frac{1}{8}$  to a  $\frac{1}{4}$  object glass in good lamp-light will show the poivrette cells glittering with a bluish white light, while the cortical cells of pepper, even if bleached, show a yellowish-white glitter. The final examination should be made with a  $\frac{1}{8}$ , or better  $\frac{1}{6}$ , in good daylight without polariser.

\* By kindness of Editor of "*English Mechanic*."

Following up a hint received from a friend, I have found the following an easy method of demonstrating the presence of poivrette to an unskilled person. Boil a portion of the sample in water to which a little caustic alkali has been added, dilute the thin mud with much water, allow to stand, and pour off the liquid from the remaining solid particles, and wash two or three times by decantation. The yellow particles are poivrette, the dark particles are husk. If any particles of bleached husk cells slightly resemble the poivrette, they may be distinguished by their softness and by the microscope.

Liverpool, 4th February, 1887.

*Conclusion of the Society's Proceedings.*

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#### ESTIMATION OF MANGANESE BY PRECIPITATION WITH MERCURIC OXIDE AND BROMINE.\*

By C. MEINEKE.

In his excellent treatise on the separation and estimation of manganese Volhard gives a method for the precipitation of the same by means of mercuric oxide and bromine from its nitric or sulphuric acid solution. His process, however, has attracted little notice. He described and explained it as follows:—The precipitation of manganese as peroxide by chlorine or bromine is accompanied by the formation of hydrochloric or hydrobromic acids, which would again dissolve the peroxide if no alkaline acetate were added. The liberated acetic acid is, however, not strong enough to prevent precipitation of other oxides, for instance, manganous oxide. If, however, a solution of manganous sulphate or nitrate in nitric acid is heated with argentic nitrate, and mixed with chlorine or bromine, all the manganese separates as peroxide. It, of course, contains argentic chloride or bromide, but is perfectly free from other metals.

A solution of mercuric chloride evolves no chlorine when heated with sulphuric acid and manganic peroxide. Instead of using silver we may therefore use the mercuric nitrate, which has the advantage of not forming an insoluble chloride. The not too dilute solution of the manganese solution is mixed with nitric acid and heated to boiling with a little pure mercuric oxide, then mixed with chlorine or bromine water till the fluid turns reddish. This is, however, not always readily noticed. To see if precipitation is complete allow to settle, and see whether more chlorine water will produce any further turbidity. A solution containing in 50 c.c. about .5 grm. of manganous sulphate, boiled with 20 c.c. nitric acid and about 1 grm. of mercuric oxide, is generally completely precipitated by the bromine in twenty minutes. If copper, cobalt, nickel, zinc, alkaline earths, or alkalies were present the precipitate must, after filtering off, be redissolved and once more precipitated. The peroxide is either ignited and weighed as manganoso-manganic oxide, or dissolved in hydrochloric acid and evaporated with sulphuric acid, and finally weighed as manganous sulphate. Volhard gives the following test analysis: 50 c.c. of a solution of manganous sulphate, containing .2615 gramme, to which was added .2 gramme of cobaltous sulphate, gave in two experiments .2618 and .2610 gramme of manganous sulphate, which proved to be perfectly free from cobalt.

My own experiments have been conducted with weaker solutions, such as would

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\* Repertorium der Anal. Chem.



fairly represent a filtrate obtained during commercial analysis. The solutions measured about 250 c.c. and contained '0933 and '3732 manganese. After acidifying and adding mercuric oxide, then boiling with bromine, the liquid remained at first quite clear, and only after prolonged boiling a slight turbidity formed. If I added coarsely powdered mercuric oxide this got coated with manganic peroxide. However, the separation was not complete until a real excess of mercuric oxide was added, and the acid completely neutralised.

Further experiments showed conclusively only mere traces of zinc are co-precipitated with the manganese, but it is of the greatest importance that the mercuric oxide and the bromine are free from iron; then this metal has a great tendency to combine with the peroxide, and even strong nitric acid fails to remove it.

To exactly ascertain the quantity of co-precipitated zinc I took 15 c.c. of solution of manganous nitrate, containing '0933 of the metal, added some zinc nitrate, diluted up to 250 c.c., heated to boiling, and added lixiviated mercuric oxide and bromine water until the liquid appeared faintly red. After decolourising with a few drops of spirit the precipitate was filtered off and washed. A portion of it obstinately adhered to the sides and was dissolved in a little hydrochloric acid, and treated with bromine water and ammonia. The precipitate was collected and washed separately, but then added to the chief precipitate. The whole was weighed as manganoso-manganic oxide. The following results were obtained:—

First experiment. Plenty (but unknown quantity) of zinc nitrate.

Impure manganoso-manganic oxide	..	..	..	..	'1332
Including ferric oxide	..	..	..	..	'0020
„ zinc oxide	..	..	..	..	'0015
Difference	..	..	..	..	'1297 = '0934 pure manganese.

Second experiment. After addition of 7.5 grammes zinc nitrate.

Impure manganoso-manganic oxide	..	..	..	..	'1348
Including ferric oxide	..	..	..	..	'0024
„ zinc oxide	..	..	..	..	'0020
Difference	..	..	..	..	'1304 = '0938 manganese.

In the meanwhile I had noticed the precipitate was scarcely affected by dilute nitric acid. In another experiment I therefore strongly acidified with nitric acid and kept boiling for five minutes, then neutralised the free acid. As the solution kept reddish no manganese could be in solution (except the trace of the permanganate). If it had turned colourless I would have been obliged to add a little more bromine and mercuric oxide.

Used 30 c.c. of the same solution and 17.5 grammes of zinc nitrate.

Obtained impure manganoso-manganic oxide	..	..	..	..	'2630
Including ferric oxide	..	..	..	..	'0015
„ zinc oxide	..	..	..	..	'0008
Difference	..	..	..	..	'2607 = '1877 manganese, or '0938 in 15 c.c.

The addition of the excess of nitric acid, therefore, practically prevents the co-precipitation of the zinc.

The same manganese solution was used for further experiments, which were made to

settle the question, whether the manganese was really precipitated as pure peroxide, and could be estimated by determining the quantity of oxygen it contained.

The best reducing agent for manganic peroxide appears to be oxalic acid, as this can be got absolutely pure, keeps for any length of time if mixed with a *mineral* acid, and can be accurately titrated with permanganate. When I treated the precipitated peroxide, first with nitric acid, to remove excess of mercuric oxide, and then with oxalic acid, I was disappointed to notice a white precipitate, which gradually disappeared on adding the permanganate, but still proved a nuisance. This compound is mercuric oxalate, manganic peroxide chemically combining with some mercuric oxide. The sulphuric acid dissolves this, and the oxalic acid now precipitates the insoluble oxalate. I therefore mixed the oxalic acid with hydrochloric acid instead. And there is not the slightest objection to this if this acid is not used in excessive quantity. For every one gramme of oxalic acid I add 2 c.c. hydrochloric acid of 1.19 sp. gravity. In titrating back the excess of oxalic acid I add 10 c.c. dilute sulphuric acid (1—1), and 20 c.c. of a 5 per cent. solution of manganous sulphate. The end reaction is then very distinctly visible.

The precipitated manganic peroxide was carefully rinsed off the filter, back into the beaker, and the filter was moistened with some of the oxalic acid (which was almost superfluous), then in turn well washed with water. In two experiments the following results were obtained:—

15 c.c. of the solution containing .0933 gramme gave a precipitate absorbing .2152 oxalic acid = .0939 gramme.

60 c.c. of the same solution = .858 oxalic acid = .3745 gramme.

It is, therefore, plain the manganese really precipitates as pure peroxide, which further test analyses will prove.

(To be continued).

## NITRITES AND NITRATES.

By A. PERCY SMITH, F.C.S., F.I.C., RUGBY.

In the *Chemical News* for January 23, 1885, there is a paper, by Mr. R. Warrington, on the various methods of detecting nitrous acid, and he shows that Griess' naphthylamine test is the most delicate of all, being able to detect one part of nitrogen in 100 millions of water.

I have lately made some experiments, in order to ascertain whether the total nitrogen, as determined by the copper-zinc couple, agrees with the sum of the separate estimations of nitrogen as nitrites and nitrates.

The naphthylamine test is one of great delicacy, and requires great care in its application. It is made as follows:—

To 100 c.c. of the water add five drops of a saturated solution of sulphanilic acid, three drops dilute hydrochloric acid, and three or four drops of a saturated solution of naphthylamine hydrochlorate; mix, and allow to stand. In cold weather the water



should be slightly warmed. A pink colour will make its appearance in the liquid, forming slowly or quickly, according to the amount of nitrite present. In cases of extreme dilution the maximum effect is not developed in less than half-an-hour. Care must be taken not to acidify too strongly, as in such case *nitrates* give a colour reaction with the naphthylamine. Amines form nitro-compounds with great facility.

To determine the amount of nitrogen represented by the tint produced, take 10 c.c. of a solution of pure potassic nitrite, containing 0.1 milligram. N, dilute to 100 c.c. with *pure* water, and apply the test as directed. It will be necessary to make a blank test with the distilled water, which frequently is sufficiently impure to give a decided colouration.\* It is impossible to take too many precautions respecting the cleanliness of the apparatus employed. On one occasion I used a measuring tube which had been standing on a shelf for some time. I rinsed it thoroughly at the sink, but the colour developed in that tube was three times as intense as that given by the same water in another tube. After cleansing the tube with acid, a fresh determination produced a correct result.

The standard nitrite solution is prepared according to the directions given in the ANALYST, vol. VI., p. 38, viz., from silver nitrite decomposed by potassic chloride.

In comparing the intensity of colour in the two tubes it is not sufficient merely to reduce the length of the column of the darker liquid. It is impossible by that means to arrive at any agreement in tint. The darker solution must be poured into a measuring glass, and successive additions of water made, until 100 c.c. (poured back into the comparing tube) equal in tint the 100 c.c. of solution with which it is compared.

I estimate nitrates by the phenolsulphonic acid method described in my paper, ANALYST, vol. X., p. 199. In that paper I have directed five drops of the acid to be used. This amount may be with advantage increased. It is necessary to have liquid enough, when the dish is tilted, to run over the residue left. The dish should, at the same time, be heated. The ammonia must not be added until the yellow colour is fully developed.

In comparing the colour produced with that of the standard solution, it is preferable to pursue the same method employed for the nitrites determination, although it is quite possible to arrive at a correct conclusion by reducing the quantity of the darker liquid. Still, mistakes may be made, owing to difference in distance from the eye, and the amount of liquid which refracts the light. It is far the better plan to compare equal volumes.

The water I experimented upon contained, by copper-zinc method, 4.316 mg. total N per litre.

200 c.c. were evaporated to dryness in a platinum dish, and gave 3.07 mg. N, as nitrates, per litre.

100 c.c. were tested for nitrites, and gave 1.25 mg. N per litre.

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\* I have found that ordinary water, kept in an open cistern, is superior to distilled water for this purpose.

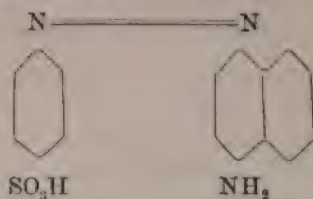
Total N, {					{ N as $N_2O_5$	3.07
4.316 }	..	..	..	..	{ N as $N_2O_3$	1.25
						<hr/> 4.32

It is probable that the  $N_2O_5$  is in *greater*, and the  $N_2O_3$  in *less* than the proper quantity, since it is impossible to acidify nitrous without forming *some* nitric acid, and the evaporation necessary in the nitrate determination may have an analogous effect. Future experiment must decide how far this is, or is not, the case.

Medical officers of health would find the naphthylamine test useful to carry with them when inspecting sources of water supply. Three small bottles and a dropping-tube would go comfortably into a waistcoat pocket. The water needs no concentration. The chemicals are inexpensive. The quantity of the reagents used must depend upon the volume of water tested. If a small test tube is used, one drop of each is sufficient.

The final result of the action of sulphanilic acid and  $\alpha$  naphthylamine upon nitrous acid is the formation of

Azo- $\alpha$ -amidonaphthalin-parabenzolsulphonic acid

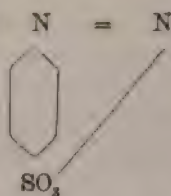


The stages of the reaction are:—

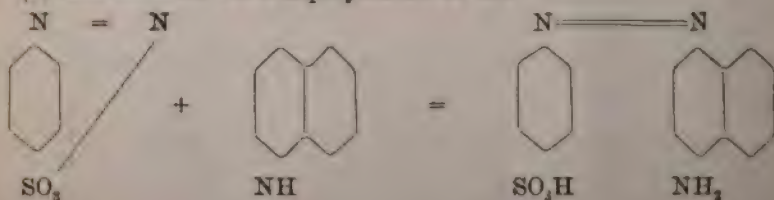
(1) The action of nitrous acid on sulphanilic acid,



gives paradiazobenzolsulphonic acid



(2) The action of the  $\alpha$  naphthylamine on this





## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

**FAT ANALYSIS.** M. KRETZSCHMAR. *Chem. Zeitung.*, vol. 10, p. 1556.—In estimating the fat in milk, or other fat-containing liquids, by the method of evaporating to dryness a weighed quantity, mixed with gypsum, in a porcelain or platinum dish, and extracting the powdered residue in an extraction apparatus, some difficulty is, as a rule, experienced in the removal of the dry mass from the dish. The author recommends the following simple method of obviating this difficulty. A piece of tin foil, about 14.8 cm. square (and of a thickness, such that the whole weighs about 2.4 gr.), is laid over and pressed into the dish (if 20 c.c. of fluid are used, a dish of 8.7 cm. diam. is convenient). The right thickness of foil is rather important. A ball of cotton-wool or a similar material, the diameter of which is slightly greater than that of the dish, is employed for pressing in the foil. The edges of the foil are bent down over the sides of the dish. The gypsum is then spread out in the dish with a pestle, so as to leave a hollow space in the centre to receive the milk. This is run in, allowed to soak into the gypsum, and evaporated to dryness, without stirring up the mass. (Sand should not be used instead of gypsum, as the results obtained are too low.) When dry, the whole mass is lifted out of the dish, rubbed up in a mortar (the foil as well), and the powdered mass extracted in the extraction apparatus. F. W. T. K.

**MICROSCOPIC DETECTION OF COLOURING MATTER IN SAUSAGES.** Dr. OTTO SCHWEISSINGER. *Pharm. Centi.* vol. xxvii., p. 441.—It is a well-known fact that a number of sausage factories colour their sausages with magenta. The latter is detected by cutting up the sausage into small pieces, extracting with alcohol, evaporating the extract down, and fixing the colouring matter on wool, either directly, or after taking it up with water. In the following the author shows that this test is not sufficient, and that in cases where it does not show the presence of extraneous colouring matter the microscopic investigation of suspected portions should be resorted to. The alcoholic extract from a sausage which the author had to examine showed not the slightest trace of a red colouring matter. However, the fact that a freshly-cut surface did not get paler on drying, but retained its fine red colour was very suspicious. He accordingly pressed out a few particles on a glass slide, and on examination under the microscope portions of the tissue were observed to be of a bright red colour, similar to that of magenta, and perfectly distinct from that of hæmoglobin, for even in the perfectly dark pieces of fresh or mortified flesh the hæmoglobin has a pale and yellow appearance. Dr. Johne ascertained that in the above case it was principally the connective tissue which was coloured.

A quantity of the intensely coloured portions were treated with alcohol and amyl-alcohol, but the colouring matter could not be extracted either by warming or allowing the mixture to stand for some days. Caustic soda solution produced complete decolourisation; sulphuric acid gradually changed the deep red colour into orange. The author believes that the colouring matter is probably a tan colour. F. W. T. K.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

**METHOD FOR DISTINGUISHING CASTOR OIL FROM OTHER FATTY OILS.** PROF. DR. FINKENER. *Chem. Zeit.*, vol. 10, p. 1,500.—The experiments were carried out with the object of finding a suitable method for customs purposes. A 100 c.c. glass cylinder of 25 mm. diameter, is provided with a 10 or 60 c.c. mark (measured from the bottom). The oil is run in to the 10 c.c. mark, and the tube is then filled up to the 60 c.c. mark with spirits of wine (.829 sp. gr.). Absolute alcohol cannot be employed, but at 17.5° C. spirits of wine of .829 sp. gr. dissolves castor oil in almost every proportion, the other

oils only slightly. The tube is closed, well-shaken, and the mixture examined after standing two or three minutes. Pure castor oil gives a clear solution. But even with 10 per cent. of other oils (olive, sesam, linseed, cottonseed, rapeseed oils) a turbidity is obtained, at the normal temperature, which does not disappear even above 20° C. Another test tried was the following:—On treating pure castor oil with sulphuric acid, a product (a sulpho-acid) is obtained, which gives an almost perfectly clear solution with 40 times the quantity of water. On treating other fatty oils (such as sesam or olive oil) similarly, very milky precipitates are obtained. The method is, however, of no good, as mixtures of 80 per cent. of castor oil or 20 per cent. of olive or sesam oil, treated similarly, also give almost perfectly clear solutions.

F. W. T. K.

ESTIMATION OF COCAINE. E. R. SQUIBB, M.D. *Ephemeris*.—The coca leaves are ground into coarse powder, 20 or 30 meshes to the inch. Of this powder 100 grams. is moistened with 100 c.c. of water, containing 5 per cent. of sulphuric acid, and is packed moderately in a cylindrical percolator, and percolated to 500 c.c. with the same acid water menstruum. This percolation is best done by means of a Sprengle water pump into a flask marked at about 500 c.c., and this amount of percolate will generally exhaust the coca sufficiently, even when the leaves are of the thick Bolivian variety. A portion of the stock of the acid water used is then balanced against a portion of the stock of re-crystallised carbonate of sodium, and it will generally be found that 6 to 6.5 grams. of the crystals are required to saturate 100 c.c. of the acid water. The percolate is then measured off into a large beaker or other similar vessel, and 50 c.c. of kerosene added to it, and the whole well stirred together. Then as much of the crystallised carbonate of sodium is added as would saturate the percolate if it was all acid water, the sodium salt being added little by little with constant vigorous stirring. In order to precipitate the alkaloid completely, the sodium salt is required in considerable excess, and the difference in acidity between the percolate, with its large percentage of extract, and the acid water gives about the proper excess. The kerosene is very thoroughly and repeatedly stirred into the mixture during four or five hours of digestion, and is then separated by means of a separatory funnel or a small syphon. Then 25 c.c. of fresh kerosene is added to the percolate and the stirring, digestion and separation are repeated twice, making three washings with the kerosene. If an emulsion should form between the percolate and the kerosene it will lie next above the percolate in drawing this off, and should be drawn off into a separate vessel with the final washing. A little dry filter paper pulp, or a little asbestos or sand stirred into this emulsion will break it up so that the kerosene can be separated completely, when it is added to the larger portion. If the percolate and the kerosene be shaken together, instead of being stirred as above directed, all or nearly all of the kerosene will be made into an emulsion. On standing two or three days this emulsion will separate wholly or partially, but as the operator can never tell beforehand whether it will separate completely or only in part, it is better not to wait, but rather to separate it as directed. Then by stirring rather than shaking, the amount of emulsion is reduced to a minimum, and if the stirring be well done, the percolate is washed free from alkaloid, and this latter is practically all held dissolved in the kerosene. But if



the operator wants to be assured that he has washed out all the alkaloid from the percolate, let him add to the latter about two grams. more of carbonate of sodium and 25 c.c. of ether and again stir, or shake and separate. The residue left on the evaporation of this ether should give no cocaine impression on the tongue, nor any saturating power to a very minute quantity of very dilute acid. The 100 c.c. of kerosene holding the alkaloid in solution is then shaken vigorously in a separator three times—twice with 10 c.c. of the acid water, and the third time with 5 c.c. of water. This washes out practically all the alkaloid as sulphate, and leaves the kerosene ready for another assay. To the 25 c.c. of solution of sulphate of cocaine contained in another separator 10 c.c. of ether is added, well shaken, and then a moderate excess of crystals of carbonate of sodium. After the effervescence, this is well shaken, and the ether separated. The solution is again washed twice with 10 c.c. more of ether each time. All the ether is then collected entirely free from even a fraction of a drop of the solution, in a beaker of at least three times the capacity of the ether. From this the ether is evaporated, and the alkaloid is left in the condition of a light amber-coloured varnish on the bottom and sides. If a capsule be used instead of a beaker, or if too small a beaker be used, the ethereal solution is liable to creep over the edges and be lost. As soon as the beaker is free from the odour of ether, it may be weighed, and the tare subtracted will give the weight of cocaine obtained from the 100 grams. of coca. This weight varies, in the writer's experience, between a few milligrams., which is called 0, and .892 grms. equal to .892 p.c., the highest result yet obtained. If the beaker, with its varnish-like coating, be set aside, there will appear, in a longer or shorter time—generally within five or six hours—minute centers of crystallisation, and these will slowly extend until within 24 or 48 hours the whole varnish-like coating will be converted into a white crystalline crust. This crystallisation takes place without any discoverable change in weight when the beaker is kept on a balance sensitive to one-tenth of a milligram.

W. H. D.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

CONTRIBUTIONS TO FORENSIC CHEMISTRY. BY PROFESSOR G. DRAGENDORFF. *Pharmac. Zeitschr. f. Russland.*—The author reports the results of investigations made under his supervision with a number of drugs more or less employed.

*Detection of Chloral.*—Having previously shown that chloral hydrate may be abstracted from aqueous solutions by agitation with ether and acetic ether, Dragendorff recommends, based upon the researches of Baron Tiesenhausen, the treatment of urine, first with petroleum-benzin, then with ether, when, on evaporation of the latter solvent, the chloral hydrate is left behind. Other organic mixtures, such as the contents of the stomach, require to be acidulated with diluted sulphuric acid, and macerated for a day with three volumes of strong alcohol; the filtrate is evaporated spontaneously until the alcohol has been volatilised, when the aqueous residue will yield fat, etc., to petroleum-benzin, and subsequently chloral hydrate to ether. Blood and organs containing much

blood retain the chloral within the coagulum, in which it is best recognized by the production of chloroform on distilling with sodium hydrate. 0.005 gm. chloral hydrate may, by these processes, be readily recognized in from 75 to 100 c.c. of mixture.

The most suitable reactions for the recognition of chloral hydrate are the following:—

The dry chloral hydrate is warmed with alcoholic soda solution and a little pure aniline; the odour of isonitril is still distinct, though faint, with 1-60,000 gm. of chloral hydrate.

Heat to 50°C. the hydrate with 1 or 2 drops of concentrated aqueous potash solution, and a little naphthol; the blue colour, produced also with chloroform, is recognized with 1-24,000 gm. of chloral hydrate.

Experiments with animals show that chloral hydrate is rapidly eliminated and transformed into products which, like urochloralic acid, do not show the reactions of chloral hydrate.

*Detection of Phenol.*—Experiments were made by Dr. Woldemar Jacobson for the purpose of isolating and recognising phenol. The organic mixtures, 100 ccm., were macerated for a day with 400 ccm. of alcohol, the filtrate was freed from alcohol by distillation at a low temperature and under reduced pressure, the aqueous residue filtered, agitated with a little petroleum benzin for the separation of fat, and then repeatedly shaken with benzol, which solvent was evaporated in watch-glasses. The following reactions were employed:—

*Dragendorff's Method.*—The residue was left in contact at ordinary temperatures with solution of mercuric nitrate, containing a little nitric acid; the red colour appears in half-an-hour with 1 phenol in 100,000 mixture.

*Jacquemin's Method.*—Dissolve 3 drops of colourless aniline in 50 ccm. of water. Dilute 5 or 10 drops of this solution with 5 ccm. of water, and add sufficient solution of sodium hypochlorite (1 sodium carbonate; 1 chlorinated lime; 10 water; filter), until a distinct violet or brown colour is produced. Add of this freshly prepared mixture to the phenol, previously mixed with ammonia, until the liquid is coloured violet or brownish, when in a short time in the presence of phenol (1:50,000) the colour will change to blue, or with less phenol (1:100,000) to green.

*Landolt's Reaction.*—Cloëtta and Schar have shown that the crystalline precipitate with bromine is still obtained in solutions of phenol diluted to the proportion 1:100,000. In separating small quantities of phenol from animal matter, Jacobson obtained amorphous precipitates with bromine, which, after drying, dissolving in alcohol, and evaporating slowly, yielded the characteristic groups of needles. Minute quantities of phenol are best dissolved in little water, and then exposed to bromine vapours.

*Detection of Thalline.*—Blumenbach found that thalline is not, or only in traces, taken up by agitating acidulated aqueous solutions with benzin, benzol, chloroform, or ether, but is dissolved from ammoniacal liquids by these solvents, however only sparingly by petroleum-benzin. Distinct reactions could be obtained by this treatment with 0.001 gm. of thalline in 100 ccm. of urine; from a similar amount of blood or food-mixture the reactions were faint, but with 0.005 gm. were distinct. The green colour produced by ferric chloride is distinct in solutions 1:10,000, and still recognisable in dilutions 1:100,000. A green colour is also produced by gold chloride, silver nitrate,



chromic acid, chlorine water, and mercuric nitrate, and in acid solutions also by chlorinated lime and potassium ferrocyanide. Iodine colours it dark brown, then dingy green; platinic chloride yellowish-green; and a red colour is produced by warm fuming nitric acid, by sulphuric and nitric acids, and by sugar and sulphuric acid. The solutions of thalline, if not too dilute, yield precipitates with the usual reagents for alkaloids.

Given to animals by the stomach or subcutaneously, thalline caused a dark colouration of the urine, which contained very little unaltered thalline, but after acidulation yielded to benzin, benzol, etc., a derivative, coloured green by ferric chloride; after precipitating the phosphoric acid with a few drops of ferric chloride the filtrate yields with more ferric chloride the red colour observed by Jaksch.

*Detection of Antipyrine.*—Blumenbach recommends treating the aqueous infusions with petroleum-benzin, and after the addition of ammonia with benzol, chloroform, or amyl alcohol, when very distinct reactions will be obtained with 0.01 or 0.005 gm., and faint reactions with 0.001 gm. of antipyrine in 100 ccm. of organic mixtures.

The alkaloidal reagents produce precipitates in not too diluted solutions of antipyrine, and ferric chloride gives in neutral solution 1:1,000 a dark brown colour, 1:10,000 a light brown, and 1:50,000 a light yellow colour. Fuming nitric acid colours dry antipyrine dark red, and in liquids green, recognisable in solutions 1:10,000. The solution, mixed with concentrated sulphuric acid and warmed with a little fuming nitric acid, becomes deep red.

Observations with animals prove that antipyrine is rapidly absorbed, and for the next eighteen to twenty-four hours may be detected in the urine; but only for a few hours in the different organs. On the other hand, antipyrine was found, after putrefaction of two weeks, in all organs of animals that had been killed about two hours after swallowing the chemical, or receiving it by hypodermic injection. W. H. D.

**ESTIMATION OF PHOSPHORIC ACID IN THOMAS' PHOSPHATE.** G. LOGES. *Repert. Anal. Chemie*, No. 6, 1887.—The increasing demand for Thomas' slags for manure causes large numbers of samples to be sent for analysis. The usual process is the well-known molybdate method; but the preliminary treatment of the slag varies in many laboratories. Either oxidising agents are used—fusion, with potassic chlorate and sodic carbonate; treatment with nitric acid or nitrohydrochloric acid; or non-oxidising bodies, such as hydrochloric acid.

The use of oxidising agents gives a too high result, as the farmer wants to know the amount of real phosphoric acid, and does not attach any value to the ferric phosphide, notwithstanding the latter gradually oxidises to ferric phosphate. Even by using non-oxidising materials, a small portion of the phosphide oxidises to phosphate, but the results are much nearer the truth. The process, as it stands now, is inconvenient, as it requires the separation of the silica and the expulsion of the hydrochloric acid by nitric acid. The removal of the silica does not seem so important, but the hydrochloric acid must, it seems, be completely driven off.

The use of concentrated sulphuric acid much simplifies the analysis, as no evaporation is needed, and it gives results accurately corresponding with those obtained by the hydrochloric acid method. The details are as follows:—

Ten grammes of the phosphate flour are moistened in a porcelain dish, with as little water as possible, and then mixed with dilute sulphuric acid (1 - 1) until all the lime has become gypsum, and carbonates and sulphides are completely decomposed. The mass, which becomes somewhat solid, is now slowly mixed, with constant stirring, with 50 c.c. concentrated sulphuric acid. The action is very energetic, but there is little risk of loss by spurting. The dish is now put on a sand-bath, and heated for about half an hour, with occasional stirring, until a thin paste has formed. The temperature must be high enough to cause fumes of the sulphuric acid. The gypsum partly dissolves as acid salt, and but few black particles are noticed, probably metallic iron. After cooling, the mass is diluted with water, and introduced in a litre flask half filled with water, when a large quantity of calcic sulphate separates out. After making up to the mark, and filtering, 50 c.c. are taken for precipitation with the molybdate. It is not necessary to oxidise any ferrous iron, as there is already sufficient nitric acid in the molybdate solution, and if a blue colour forms by reduction of the molybdate, it is soon destroyed on warming. The presence of free sulphuric acid does not in the least interfere. Silicic acid can only be present in traces. From 100 c.c. of a solution prepared as described, only .0006 gramme of silica could be recovered, and as this looked brownish, it is doubtful whether it was silica at all.

## TEST ANALYSES.

Sample.	Hydrochloric acid method.	Sulphuric acid method.	Difference.
1 .. ..	23.02 .. ..	23.11 .. ..	.09
2 .. ..	19.66 .. ..	19.71 .. ..	.05
3 .. ..	23.17 .. ..	23.36 .. ..	.19
4 .. ..	19.76 .. ..	19.71 .. ..	.05
5 .. ..	15.62 .. ..	15.70 .. ..	.08
6 .. ..	19.66 .. ..	19.77 .. ..	.11
7 .. ..	15.68 .. ..	15.60 .. ..	.08
8 .. ..	19.37 .. ..	19.33 .. ..	.04
9 .. ..	18.32 .. ..	18.47 .. ..	.15

In conclusion, the author states, this process has been used in his laboratory for some time, and has considerably lessened the labour of the analyses. L. DE K.

## APPOINTMENT.

Dr. WILLIAM JOHNETONE, F.I.C., F.C.S., has been appointed Public Analyst for the Borough of St. Ives, Cornwall.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

To the Editor of the ANALYST.

SIR,—Could our medical friends give us some information in reference to a disease which has lately broken out among Analysts, and which threatens to spread rapidly? It is, apparently, infectious and incurable, and attacks only certain constitutions. It first shows itself by the eruption of the two consonants Dr. on the note headings of the individuals affected; it gradually spreads upon their door-plates, and ultimately finds its way as an appendage to their signatures. The period of incubation varies much. I have heard of a case in which this stage lasted some years. In the earlier stages some of the patients blush when spoken to, but in the more aggravated cases the blush is absent.

Am I right in believing the disease to be a variety of the American doctor fever? And should not, seeing the virulent character of the affection, the individuals who suffer from it be subjected to disinfection and quarantine?—I remain, sir, yours faithfully,

HYDROXYL.



# THE ANALYST.

APRIL, 1887.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of the Society was held at Burlington House, Piccadilly, on Wednesday, the 9th ult., the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election, and will be balloted for at the next meeting, viz. :—C. E. Cassal, public analyst for Kensington ; G. H. Ogsten, London, analytical chemist ; F. W. Stoddart, public analyst for Bristol ; Dr. Sedgwick Saunders, public analyst for the City of London.

On the ballot papers being opened, it was announced that the following gentlemen had been elected :—As members, Dr. Theophilus Redwood, F.I.C., F.C.S., public analyst for Middlesex ; Theophilus Horne Redwood, analytical chemist, London ; J. E. Stead, public analyst for Middlesborough ; A. Norman Tate, F.I.C., F.C.S., analytical chemist, Liverpool. As associate, H. Richmond, assistant to Mr. Hehner.

The President announced that he had received a letter from Sir F. Abel, on the subject of the proposed Imperial Institute, and asking him to bring it before the members of the Society individually as well as collectively. The Council had considered the matter to some extent, and they thought it would not be desirable to make an individual subscription. They were, however, in correspondence with other societies as to what course they could adopt with a view to making some joint subscription. The matter was left in the hands of the Council to use their discretion as to what was best to be done.

The President called the attention of the members to the following resolution which had been passed by the Council :—

“That the Members of the Society be requested to inform the Hon. Secs. of, and fully report to them on, any interesting cases or other important matters connected with the working of the Sale of Food and Drugs Act ; and that the Hon. Secs. be requested to draw up periodical reports to be read before the Society and published in *THE ANALYST*, embracing the points of importance in the communications made to them.”

The following papers were read and discussed :—

Experiences of Curious Impurities, Admixtures, and Substitutions, by A. H. Allen, F.I.C., F.C.S.

Further Experiments on the Volatility of Glycerine, by Otto Hehner.

The next meeting of the Society will be held at Burlington House on Wednesday, the 13th inst.

## NOTES ON THE COMPLETE ANALYSIS OF MILK.

By DR. P. VIETH, F.C.S.

*Read at the Meeting, February 9th, 1887.*

So much attention has been paid of late to milk analysis that it might appear not only superfluous but altogether impossible to say anything new on the subject. However, if one looks more closely into the matter, one still finds room for some observations which

have not been brought forward on previous occasions, and points which might be discussed with advantage. I will, with your permission, add a few remarks to the many which I have made on the subject on former occasions.

Before doing so, I wish to guard myself against the assumption that I intend reopening the question, which more than three years ago was put before and, after two years careful work and earnest deliberations, decided by the Milk Committee then appointed. The abandonment of the old and the adoption of the methods and limits proposed by the Committee was to me a great pleasure and satisfaction, and I feel convinced that if the methods proposed by the Committee are only properly carried out by every public analyst, there will be in future much more agreement in milk results, much less conflicting evidence, and consequently less ill-feeling, but an increasing feeling of greater certainty.

So far as an addition of water or abstraction of cream is concerned, the adoption of the Milk Committee's report ought to have closed the question of examining milk, if not for ever, certainly for a long time to come; but there is a good deal to be done beyond ascertaining total solids and fat, and a public analyst may be called upon any day to give his opinion on a sample of milk, a complete analysis of which being indispensable for the purpose. It is the subject of "complete milk analysis" on which I am going to make some remarks. What I, for the sake of this paper, call complete may be considered very incomplete by others, for I shall neither talk of all the bases and acids constituting the milk-ash, nor shall I take into account the small traces of urea and other bodies which are alleged to be present in milk.

*Ash.*—It is well known that when a milk residue is heated over a flame, the sugar and nitrogenous compounds present leave behind a coal, which is by no means easily burnt away. If a small flame be employed, it takes a considerable time to get a white ash, and the attempt to accelerate the process, by using a higher temperature, is invariably accompanied by a loss of chlorides. The safest way to avoid a loss is to char the milk solids, extract the soluble salts with hot water, ignite the coal completely, and then evaporate the solution of the soluble salts on the water bath. Insoluble and soluble salts may thus be determined separately. The extraction of the soluble salts is, however, not indispensable, for if igniting is carried out at dark red heat and not prolonged more than necessary, practically identical results are obtained as in the previous case, proving that under these circumstances no, or an inconsiderable loss only, takes place. Keeping the ash at bright red heat for some time has a very marked effect. In five samples of milk the ash was determined and the platinum capsules containing it afterwards exposed to bright red heat for one half hour. After that time there was a decrease in the percentages of ash from .72 to .54, .78 to .58, .70 to .56, .74 to .56, and from .74 to .58 respectively. Another experiment was made in this way, that of one and the same milk six samples were taken and the ash determined; in every case .74 per cent. was found. The first ash was put aside; the second kept at dark red heat for one half hour, when the ash decreased to .70; the third, fourth, fifth, and sixth ashes were heated at bright red heat for five, ten, twenty, and thirty minutes respectively, and the result was a decrease to .70, .60, .56, and .56 per cent. In all the six ashes the chlorine was then titrated, and found to amount to .0923, .0852, .0781, .0284, .0036, and .0036 per cent. of



the milk. These results show that heating a milk-ash at bright red heat for ten minutes involves a serious loss of chlorides, while after heating has been continued for twenty minutes, practically the whole of the chlorides was evaporated.

*Milk-Sugar.*—It has been shown by Schmoeger that on evaporating milk to dryness the milk-sugar contained in it is left behind in the anhydrous state (THE ANALYST, xi. p. 142). This makes it necessary, in complete milk analyses, to calculate the sugar as anhydrous milk-sugar, whatever the method of determination may be. Two convenient methods present themselves for the exact determination of sugar in milk, viz., titration and polarisation. I am in the habit of using the latter method, employing Mitscherlich's Half-Shadow Polariscopes, of which I gave you a short description some time ago (THE ANALYST, xi. p. 144).

In order to test the instrument, I made some solutions of milk-sugar, examined them in the polariscope, and determined the amount of solids left on evaporation. To have a further and quite independent check, I requested Mr. A. W. Stokes to be good enough to titrate the sugar in the solutions, to which request he very kindly consented. The following are the results:—

Solution No.	Evaporated		Polarised $C_{12} H_{24} O_{12}$	Titrated $C_{12} H_{24} O_{12}$
	$C_{12} H_{22} O_{11}$	$C_{12} H_{24} O_{12}$		
1.	6.84	7.20	7.5	7.48
2.	5.80	6.11	6.3	6.20
3.	4.16	4.38	4.4	4.20
4.	2.84	2.99	3.1	3.04

In four samples of milk the amount of sugar was found, by Mr. Stokes and myself, as follows:—

Sample.	Polarised.	Titrated.
1. ....	5.14	5.01
2. ....	5.35	5.23
3. ....	5.14	5.01
4. ....	5.35	5.17

For the titration, the "Pavy Ammoniacal Cupric solution" was used by Mr. Stokes. The determination by means of the polariscope gave, in every instance, slightly higher results than titration, the largest difference being .2 per cent.

*Proteids.*—No doubt can be entertained that several distinct nitrogenous compounds exist in milk, but on the number and exact nature of these bodies opinions differ very widely; for the sake of convenience, we will comprise them under the collective term of "proteids." By the addition of acid to milk, part of the proteids is precipitated, the precipitate enclosing also the fat present; this part is usually called caseine, and amounts to about 77 per cent. of the total proteids. On heating the filtrate from caseine to boiling, another precipitation, or rather coagulation, takes place, the body thus thrown down being termed albumen, and amounting to about 10 per cent. of the whole of the proteids. The remaining 13 per cent. left in the filtrate from albumen are precipitated by various metallic salts and by tannin. This precipitate undoubtedly consists of a

mixture of several compounds, but entering upon this point more closely would carry us too far for the purpose of this paper. We will term the third precipitate lacto-proteine.

The separation of the three classes of nitrogenous compounds from each other is not as exact as could be desired. If in precipitating the caseine an excess of acid is used, part of the precipitate will get redissolved; again, it makes a difference whether, for the purpose of precipitating the albumen, the filtrate from caseine is just brought to or kept boiling for some time and thereby concentrated. In case the precipitation of caseine and albumen should not have been complete, what is left of them in solution will swell the tannin precipitate. However, if working carefully, duplicate determinations agree pretty well, as is shown by what follows. In twenty-six samples the proteids were determined separately and collectively, and the following results obtained:—

Caseine, the differences in duplicates varied from .00 to .22, and averaged .048; albumen, differences from .00 to .14, average .030; lacto-proteine, differences from .00 to .27, average .054. For the collective determination of the proteids, Ritthausen's method was employed, which consists in diluting the milk with ten times its bulk of boiling water, precipitating the proteids with sulphate of copper, adding sufficient potash to nearly neutralise the liberated acid, collecting and washing the precipitate (which includes the fat) on a filter, drying it, extracting the fat, and ascertaining the combustible part of the precipitate. Duplicate determinations differed from .03 to .12, the average difference being .060. When comparing the percentage amounts of proteids obtained by adding up the figures for caseine, albumen, and lacto-proteine, with those obtained by employing Ritthausen's method, it was found that the latter method had given in seven cases a higher, and in nineteen cases a lower, percentage of proteids. The differences between the two methods varied from +.22 to -.24, and on average there was a minus of .073 on the side of Ritthausen's method. On the whole, I am inclined to put more faith in the correctness of results obtained by using the latter method.

*Fat.*—After attempting to extract the fat from the residue obtained in determining the total solids has been abandoned, I hope, by every one, and for ever, there are virtually two methods only, competing with each other, viz., the paper method and the plaster method. When employing the former it is indispensable that the paper used in the process should be most thoroughly exhausted from matter soluble in ether. As far as agreement in duplicates is concerned, both methods give very satisfactory results. Working upon sixteen samples of whey, skim milk, milk, and mixtures of milk and cream, the following results were obtained:—Plaster method, differences ranged from .00 to .21, and averaged .045; paper method, differences ranged from .00 to .12, and averaged .056. On comparing the results of the two methods it was found that in all and every case a higher percentage of fat was obtained by using the paper method, the plus ranging from .07 to .46, and averaging .271.

It has always been contended that that method is the most correct one by which the highest percentage of ether-extract is obtained. Arguing on the same line, and, moreover, considering that, when extracting milk dried up on plaster and on paper, there is no difference to be found in the composition of the fat extracted, justifying the opinion that the latter was not pure butter-fat in both cases, the laurel should be awarded to the paper method. There is one point, however, which makes the case



somewhat peculiar. The old methods, which were in use until some years ago, were denounced as inadmissible, because the solid, horny nature of the milk residue did not allow the solvent to penetrate the whole mass and remove the total of the fat present. The necessity was pointed out to break up the milk residue, and spread it over a large surface by adding an indifferent powder, such as glass, sea-sand, or, preferably, plaster, to the milk and then dry it. It was further contended that this course was the more necessary the less fat there was present, and the work of the Milk Committee has borne out that the results of different methods always agree better when working on material rich in fat, while with poor skim milks the widest differences were obtained. Now, for the paper method, it is also claimed that the higher results are due to spreading the solids out on the largest surface which is practically applicable; but on comparing the results with those yielded by the plaster method, the largest differences are not found with poor milk, but, just on the contrary, with the increasing richness of the material the differences likewise increase. Among the sixteen samples operated upon there were five containing less than 1 per cent. fat; the average difference in favour of the paper method was .204; in seven samples, containing from 1 to 4 per cent. fat, the average difference was .243; and in four samples containing from 4 to 9 per cent. fat, the average difference was .405. One should be inclined to think that there can be no difficulty whatsoever to extract the fat to the last trace by any—even the Wanklyn—method from milk solids consisting of about 50 per cent. fat, and it is difficult to perceive why it should make a difference, whether such a milk is dried up with plaster, or on paper. I am sorry to say that I have not been successful in my endeavours to find an explanation for a point which certainly wants clearing up.

*Water.*—The determination of the water, or what comes to the same, the total solids, has not given occasion for much discussion, it being almost generally admitted that drying the solids to constant weight at the temperature of boiling water in a dry atmosphere is the only admissible and perfectly correct method. And, still, doubts might be entertained whether it is possible to rid the solid, horny residue of the last traces of moisture in this way, and whether not in this case, too, a spreading over a large surface be useful, or even necessary. To get some information on this point, I determined in ten samples of milk the solids; four determinations were made in each sample, two in the usual way, and two with the addition of annealed sea-sand in sufficient quantity to suck up the milk. The differences in the duplicates made without sand varied from .00 to .14, and averaged .066; in the samples dried up on sand the differences ranged from .00 to .08, and averaged .032. Comparing the results of the two series showed that in four cases there was a plus, in six cases a minus, on the side of the samples dried with sand, the differences ranging from + .06 to - .10, and averaging - .023. There was then, practically speaking, no sensible effect produced by drying up on sea-sand. I may mention that these experiments were made six years ago. The question has been raised once more when the paper method was made known, and it was contended that by this method alone, and after the extraction of the fat, a thorough drying of the non-fatty solids can be effected. I must admit that I could never get myself to believe that it is of advantage to weigh and re-weigh a pretty large piece of blotting paper, which is coiled up, repeatedly extracted and dried, and thereby a good deal handled. I have

made a very few comparative experiments only—in fact, my experience is limited to four samples. Total solids obtained by the paper method differed in duplicates from .01 to .08, the average difference being .035; usual method gave differences from .03 to .09, the average being .050. When comparing the results of the two series, it was found that the paper method gave in one instance very slightly more, in three instances less total solids, the differences ranging from + .03 to - .34, and averaging .153. My experience being limited to such a small number of samples, I must abstain from commenting upon the results.

*Complete Analyses.*—I will now draw your attention to a series of complete analyses. In order to have materials containing the component parts of milk in the most varied proportions, I operated on whey from skim milk, and from mixtures of milk and cream, further, on skim milk, whole milk, and milk to which cream had been added. The methods used for the several determinations were the following:—Water, drying solids to constant weight; fat, plaster process; proteids, precipitated with sulphate of copper; sugar, polarised; ash, soluble and insoluble parts, determined separately or collectively by most careful ignition. All the figures are means of well agreeing duplicates.

Water.	Fat.	Proteids.	Sugar.	Ash. { Soluble. Insoluble. }		Total.	Remarks.
92.92	.09	1.17	5.38	.51		100.07	Whey from skim milk.
90.91	2.44	1.23	5.10	.48		100.16	" " milk + cream.
85.16	8.53	1.38	4.78	.43		100.28	" " " "
90.77	.19	3.44	5.00	.26	.52	100.18	Skim milk
90.64	.17	3.48	5.20	.24	.52	100.25	" "
90.62	.21	3.56	5.11	.25	.54	100.29	" "
90.27	.31	3.83	4.85	.78		100.04	" "
90.11	.36	3.71	4.96	.80		99.94	" "
88.07	3.22	3.15	4.97	.24	.51	100.16	Milk
87.69	3.31	3.32	5.16	.29	.54	100.31	"
87.52	3.63	3.33	5.05	.26	.52	100.31	"
87.34	3.60	3.41	5.15	.74		100.24	"
87.28	3.86	3.29	4.75	.22	.54	99.94	"
85.14	5.78	3.53	4.88	.69		100.02	Milk and cream.
84.93	5.86	3.31	5.09	.75		99.94	" "
82.28	8.65	3.49	5.10	.69		100.21	" "

If the fact that the percentage amounts of the several component parts sum up exactly or close to one hundred is to be taken as a proof that the individual determinations, and consequently the whole analyses, are correct, the preceding analyses leave little to be desired, and larger percentages of fat and water would only tend to destroy their apparent accuracy. We all know,



however, that neither analysts nor analytical methods are infallible, and that it is of not unfrequent occurrence that an error in the one direction is counterbalanced by another in the opposite direction. Thus if, in the case of the analyses put before you, which on average sum up to 100·15, the fat ought to be higher by ·25, and the water by ·15 per cent, ·55 per cent. too much must have been found in the other three, or if we exempt the ash, two determinations. As long as I cannot see with which determination the error rests, you will, I dare say, not think me very wrong if I stick to methods which have given such satisfactory results as I have been able to bring before you.

### ON THE NON-VOLATILITY OF GLYCERINE WITH AQUEOUS VAPOUR.

By OTTO HEHNER.

*Read at the Meeting, March 9th, 1887.*

I BROUGHT, at the last meeting of the Society, some evidence before you as to the non-volatility of glycerine with aqueous vapour, in the case of dilute solutions (ANALYST, XII., p. 44). It was there shown that on boiling down 300 c.c. of water containing ·3 grms. of pure glycerine to about 150 c.c.—that is, on concentrating a ·1 per cent. solution to ·2 per cent.—no loss of glycerine could be traced, and that consequently the washings from fatty acids could be safely freed from alcohol without fear of loss of glycerine, provided the percentage of glycerine in the fluid was as low as above indicated.

I have now studied the behaviour of more concentrated solutions of glycerine, with results which are at direct variance with our preconceived ideas. These are mainly based upon experiments by Nessler and Barth (Zeitschr. f. Anal. Chem., XXI., p. 44, and XXIII., p. 329, in abstract Allen, Com. org. analysis, 2nd edit., Vol. II., p. 284 foot-note). These gentlemen ascertained the loss (T) sustained by weighed quantities of *dry* glycerine on being heated for one hour on a waterbath, and that of glycerine solutions, of various strength, which were evaporated on the water-bath, the residue being kept for one hour at the same temperature, after a syrupy consistence had been reached (W + T). By subtracting T from W + T they obtained W, which they called glycerine volatilised with aqueous vapour. They found this loss to be on evaporating:—

50 c.c. of a 2 per cent. solution 4·9 per cent. of the glycerine.					
25	"	2	"	3·4	"
100	"	1	"	5·1	"
50	"	1	"	2·7	"
25	"	1	"	2·3	"
100	"	·5	"	2·4	"

Nessler and Barth conclude from these results that the loss of glycerine depends upon two conditions, the strength of the solution and the volume of water evaporated.

It is, however, easy to see that such evaporation experiments cannot afford any guidance; for at no time of the experiment has the solution a definite strength; it increases continuously as the water escapes, and no information is obtained at what concentration the glycerine really does commence to go away. It is impossible to say when a syrupy residue has been obtained; hot glycerine indeed is not syrupy at all, and possibly the total loss occurs at the highest stages of concentration. According to the

figures of Nessler and Barth, the loss increases, with the same solution, with the quantity of solution evaporated. Thus if 25 c.c. of a 1 per cent. solution lose 2·3 per cent., and 50 c.c., 2·7 per cent., and 100 c.c., 5·1 per cent., then on evaporating a sufficiently large volume, say 2 or 3 litres, no glycerine should be left at all, and it would obviously be impossible to regain any glycerine whatever by the concentration of soap liquors, from which, in practice, there is no difficulty in recovering the glycerine in almost theoretical quantities.

Nessler and Barth's results show solely that, on evaporating glycerine solutions to dryness, glycerine is lost at some stage of the proceedings.

To ascertain at what point of concentration glycerine was thus volatilised, I kept solutions of glycerine of known strength vigorously boiling in a covered beaker, over the naked flame, continually replacing the evaporated water, so that the solutions were always as nearly as was practicable of the same strength. The glycerine employed had a specific gravity of 1·2572, corresponding to 95·55 per cent. of pure glycerine. All estimations were made according to the chromate method described by me in ANALYST, XII., p. 44.

1. 8546 grms. of glycerine, corresponding to 8164 pure glycerine, were dissolved in 50 c.c. of water. The fluid was boiled for many hours, then diluted to 100 c.c., and 25 c.c. oxidised with chromate. Found 20027 grms., or 98·1 per cent. of the glycerine taken.

Duplicate experiment: 98·3 per cent. of glycerine.

2. 32721 grms., corresponding to 31265 grms. pure glycerine, were diluted to 100 c.c., and the solution boiled vigorously for two hours, during which time 176 c.c. of water were evaporated. The solution was then made up to 250 c.c., and 25 c.c., containing originally 31265 pure glycerine, were oxidised. Obtained 3117 or 99·7 per cent. of the quantity originally taken.

3. 78285 grms., equal to 74801 pure glycerine, in 100 c.c. water, were boiled for two hours, 222 c.c. water evaporating. The fluid was then brought to 250 c.c., and 10 c.c., corresponding originally to 2992 grms. pure glycerine, were oxidised. Found 100·3 per cent. on the original quantity.

4. 127436 grms., equal to 121765 grms. pure glycerine, in 100 c.c. were boiled for two hours, 229 c.c. water evaporating. The fluid was then diluted to 250 c.c., and 10 c.c. were twice treated, containing 4871 pure glycerine. Obtained 48809 grms., equal to 100·2 per cent., and 48995 grms., corresponding to 100·6 per cent. of the original glycerine.

5. 290124 grms., containing 277213 grms. pure glycerine, made up to 100 c.c., and boiled for two hours, 235 c.c. of water evaporating. The fluid, made to 250 c.c. had a specific gravity of 1026·4; hence the 250 c.c. weighed 256·6 grms.; of these 33248 grms., corresponding to 3595 of glycerine, were oxidised. Found 36281 grms., or 100·9 per cent. Again, 21124 grms., equivalent to 2282 grms. glycerine, yielded, on oxidising, 23007 grms., or 100·8 per cent. of the glycerine taken.

6. 513684 grms., with 490825 grms. pure glycerine, were boiled as above, previously made up to 100 c.c.; 257 c.c. water evaporated. The fluid, made up to 250 c.c., had a specific gravity of 1046·9, hence 250 c.c. weighed 261·7 grms., of this 24364 grms.,



corresponding to 4569 grms. pure glycerine, yielded, on oxidation, 45956 grms., or 100·6 per cent. of glycerine.

7. 77·3096 grms., with 73·8693 grms. pure glycerine, were made up to 100 c.c., and boiled for two hours. 295 c.c. water evaporated during that time. The fluid was then diluted, so that it weighed 267·55 grms. Of this 7934 grms., corresponding to 21905 grms. of the original glycerine, were oxidised. Found 2166 grms., or 98·4 per cent. of the original amount.

In a duplicate experiment 8636 grms., equal to 23843 of glycerine, were oxidised; 98·8 per cent. being found.

It appears to my mind completely established by these experiments that glycerine is *not* volatilized with aqueous vapour from any solution containing less than 50 per cent. of glycerine. Even in a 73·8 per cent. solution the loss, if any, is exceedingly insignificant. Whatever loss, therefore, takes place on evaporating glycerine solutions must occur when the residue is nearly or quite anhydrous. It must not be overlooked, also, that in my test experiments the solutions were kept at their boiling-point, which, in the case of the stronger fluids, was considerably above 100°; the 73·8 per cent. solution, for instance, boiled from 115° to 116°; the escaping steam was at 100° C. Hence, if at 115° the glycerine was practically non-volatile, it may be safely assumed that at 100° no loss whatever would have occurred.

It may be desirable to establish the exact degree of concentration at which volatility commences, but this could not well be done in the way which I have followed. It is too difficult to keep the water level absolutely constant, and in the case of very strong solutions the slightest variation would represent wide limits of variation in the concentration. Besides a decided browning of the liquor takes place on boiling strong solutions for two hours, indicating some degree of decomposition.

*Conclusion of the Society's Proceedings.*

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## ON LONG PEPPER.

BY PROFESSOR J. CAMPBELL BROWN, D.Sc.

In a valuable paper published in Vol. XI. of THE ANALYST, Mr. Heisch remarks, "How far an admixture of long pepper is to be considered an adulteration, I cannot say." This seems to be the general attitude of analysts at present, although some have adopted a decided tone.

It is now time that all should take up a decided position in regard to this form of adulteration. Long pepper is the fruit of *Charica Roxburghii*, and does not consist merely of the berries analogous to the pepper-corns of the true pepper-plant; it bears much the same relation to them that wild grass-seed would bear to oatmeal. It consists of the small berries with the husks and indurated coverings hardened together and to the central woody stem, much in the same way that in pines the seed and coverings are all hardened into one cone. Long pepper is for the most part derived from wild plants of *Charica Roxburghii*, which grow by the sides of the water-courses in India.

Consequently, it always brings with it a mass of dirt, picked up from the soil of the banks whereon it grows, imbedded in the crevices and irregularities of the fruit, which dirt the native collector takes care not to lessen, but rather to increase, seeing that he is paid by weight for what he brings down to the merchants.

In commerce we find accordingly that it has always from three to seven per cent. of insoluble sand and clay, in addition to the proper ash of the fruit. And it is difficult, if not impossible, to clean long pepper before grinding, in the way that true pepper can easily be cleaned; it can with difficulty be cleaned by hand.

The ash contains a very large proportion of salts insoluble in hydrochloric acid. When ground, the hard husk and woody centre, as well as the dirt, are necessarily ground along with the minute berries. The ground long pepper contains not only sand, but more woody fibre than ground genuine pepper of the corresponding shade, although not so much total cellulose as the most husky black pepper. It has the composition shown by Mr. Heisch in his paper. I can confirm his results by the following

#### ANALYSES OF LONG PEPPER.

##### CAREFULLY CLEANED BY HAND.

	Total Ash.	Sand and Ash insoluble in HCl.	Total matter Soluble in 10 per cent. HCl.	Starch and matters convertible into Sugar.	Albuminous matter soluble in Alkali.	Cellulose.	Extracted by Alcohol.	Extracted by Ether.	Total Nitrogen.
1	8.91	1.2	67.83	44.04	15.47	15.7	7.7	5.5	2.1
2	8.98	1.1	68.31	49.34	17.42	10.5	7.6	4.9	2.0
3	9.61	1.5	65.91	44.61	15.51	10.73	10.5	8.6	2.3

Although the cost of long pepper is at present nearly as high as some very inferior varieties of black pepper, yet the price is generally decidedly lower; even now long pepper is much cheaper than the pepper with which it has been sometimes mixed of late, and its use affords a handsome illegitimate profit, to the detriment both of the grocer and his customer. Long pepper has been, and is, legitimately used for pickles, but it is not known, nor has it been recognised by the trade, as ground long pepper; and all the respectable grocers, and others of whom I have inquired, say decidedly that they would not buy, nor retain if received, any ground pepper which they knew or suspected to contain an admixture of long pepper. In fact, it is no more right to give pepper containing long pepper in response to a request for simply "pepper" than it would be to give horse-chestnuts instead of Spanish chestnuts in response to a request for simply "chestnuts." It may, of course, be sold as ground long pepper, without offence; but no one would buy it. Not only is long pepper a fraudulent admixture in ground pepper, but it is objectionable on the score of quality and flavour. Its disagreeable, offensive



flavour is developed by warmth. Any candid person can convince himself of the real cause of the objections which housekeepers and grocers alike have to ground long pepper if he will heat up a piece of cold meat between two plates, and sprinkle some fresh long pepper on it; the smell and flavour are so offensive that he will feel obliged to reject the meat.

Much of that which one gets whole in shops is very old, and has lost much of its flavour and strength, so small a sale does it command.

The presence of long pepper in ground pepper may be determined by the following characters:—

1. Colour. If any serious quantity of long pepper is ground in with the ordinary pepper it imparts some of its peculiar slaty colour; but this is made much lighter by the now very common practice of sifting out much of the darker or husky portions of the long pepper before mixing it with the genuine pepper. Bleaching is also resorted to, but not hitherto very effectively.

2. The odour of the mixture when warmed is unmistakable by an educated olfactory sense, even if the quantity is comparatively moderate. Attempts are made to disguise the odour by bleaching, but this has not been successful. The ethereal extract also, and even the alcoholic extract from which the solvent has been evaporated at a low temperature, yields when warmed the characteristic odour very plainly.

3. Long pepper introduces sand into the pepper with which it is mixed, often to a considerable amount. If the pepper is white this has more importance than has hitherto been accorded to it; for white pepper does not contain even as imported  $2\frac{1}{2}$  per cent. of sand, and any white pepper containing so much sand must have had the sand improperly introduced, either by direct mixing of Calais sand, or in some other way.

Long pepper from which the husk particles have been sifted out when added to white pepper invariably introduces its sand along with it, as well as some spent bleach, attempts have been made to bleach it.

4. The woody matter in ground long pepper is always considerable, arising both from the smallness of the berries compared with the hardened setting, and from the central woody tube. This may be detected either by chemical analysis or by the microscope, and some of it by the naked eye or a large hand lens.

If the sample is spread out in a smooth thin layer on strong paper, by means of an ivory paper-knife, pieces of fluffy woody fibre will be detected, especially if the smooth thin layer be tapped lightly from below. Those pieces come from the central part of the indurated catkin which cannot be completely ground fine, as genuine pepper stalks are, and are very characteristic if carefully examined. Much of these are of course removed by the grinders' sieves; but enough finds its way through the meshes of the sieve to be useful as a corroborative indication.

5. Particles of husks if present can be distinguished from genuine pepper husks.

6. A proportion of the starch granules of long pepper are of larger size, above  $0.002$  inch, and of angular shape, very slightly smaller than rice granules, and more closely aggregated in clusters, or isolated.

Here it is necessary to notice that the statement is made in books that genuine pepper starch is round in form. Pepper starch is doubtless round in the main, but not

invariably. The loose granules of the interior are spherical, but in the denser portions of the berry they become more angular by pressure on each other.

University College, Liverpool.

#### DETECTION OF ARTIFICIAL COLOURING MATTER IN BUTTER, OLEOMARGARINE, FATS, OILS, ETC.

BY EDWARD W. MARTIN.

DURING the year 1884, at the request of Hon. J. K. Brown, New York State Dairy Commissioner, a number of experiments were made for the purpose of arriving at a rapid method for the detection of artificial colouring materials in oleomargarine. This was a matter of some importance, as one section of the New York law forbids the colouring of oleomargarine to resemble butter, and prosecutions were made on this point alone.

Many methods had been used up to this time, but all were more or less complicated, and the amount required in some instances rendered them inconvenient and sometimes inapplicable. The great obstacle in separating colouring matter from fat is that the reagents which dissolve the colouring matter also dissolve the fat; furthermore treatment of the fat with a reagent in which it is insoluble fails to extract the colouring matter.

I found, however, that when annatto was present, if the filtered pure fat from oleomargarine or butter was dissolved in bisulphide of carbon, chloroform, ether, kerosene, etc., and to this solution water made slightly alkaline with caustic potash or soda was added, that the alkaline water dissolved out the colouring matter. Moore (*ANALYST*, XI., 163), found that carotin could not be detected in this way, but by substituting for the alkaline water an exceedingly dilute solution of ferric chloride, the presence of carotin could be shown.

These tests, however, are limited to annatto, turmeric, and carotin. In the course of further experiments, I found that the following method seemed to detect not only these three colours but other yellow colours, such as the aniline and naphthol yellows, and possessed the additional advantage of being applicable to the butter or oleomargarine directly, not previously freed from water, curd, and salt.

The following is the test:—

Dissolve 2 parts of bisulphide of carbon in 15 parts of methylalcohol by adding small quantities of the bisulphide to the methylalcohol, and shaking gently; 25 c.c. of this mixture are placed in a convenient tube, 5 grammes of the butter, fat, or oil added, and the tube shaken. The bisulphide falls to the bottom of the tube carrying with it the fatty matter, while the colouring matter, if any is present, remains in the methylalcohol. The separation takes place in one to three minutes. Instead of methylalcohol other solvents may be used, such as ether, alcohol, acetone, etc.

If the amount of colouring matter present is small, more of the fat can be used.

I have made numerous experiments, and in no case did the test fail if artificial colouring matter was present, while the natural colours of fats, oils, butter, or oleomargarine in no instance coloured the methylalcohol.

*Laboratory of Professor E. Waller,*

*School of Mines, Columbia College, New York.*



## AMOUNT OF ASH IN AMERICAN TALLOWES.

By WALTER LEE BROWN, CHEMIST, CHICAGO, BURLINGTON AND QUINCY RAILROAD CO.

As an appendix to the article which appeared in the August, 1886, number of THE ANALYST, entitled "American Method for the Testing of Tallow for Railroad Use," the following observations on the per centage of ash in American tallowes may prove of some interest. The figures were obtained as the results of using the process for detecting soap, given in the above article, *i.e.*, by noting the amounts of ash.

From January 15, 1886, to January 25, 1887, 59 lots, representing 768 barrels, had their average ash determined. The number of samples (or barrels) in each lot ranged from 1 to 35, usually from 10 to 16.

The following table shows the number of lots of each grade ("refined" or "common butchers' stock") which gave the various percentages indicated:—

Per Cent. Ash.	Grade.	No. of Lots.
None .. ..	Refined .. ..	10
" .. ..	Common butcher's stock .. ..	3
" .. ..	Doubtful .. ..	2
0.001 .. ..	Common butcher's stock .. ..	3
0.002 .. ..	Refined .. ..	4
" .. ..	Common butcher's stock .. ..	9
" .. ..	Doubtful .. ..	1
0.004 .. ..	Refined .. ..	6
" .. ..	Common butcher's stock .. ..	6
" .. ..	Mixed .. ..	1
0.006 .. ..	Common butcher's stock .. ..	4
0.008 .. ..	" .. ..	2
0.010 .. ..	" .. ..	2
0.012 .. ..	" .. ..	1
0.024 .. ..	Refined .. ..	1
" .. ..	Common butcher's stock .. ..	1
0.028 .. ..	" .. ..	1
0.032 .. ..	" .. ..	1
0.046 .. ..	Refined .. ..	1

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59

The latter one should have been investigated.

Average Ash of the 59 lots = 0.0048 per cent.

" " " Refined Tallow = 0.0046 per cent.

" " " Common butcher's stock = 0.0061 per cent.

The three doubtful and the one mixed lot are not considered in the last two averages.

If the two samples running 0.024 and 0.046 per cent. of the refined tallowes are thrown out as abnormal, the remaining 20 lots will average 0.0016 per cent.

Of the 59 lots, 45 averaged under 0.006 per cent., or with a general average of 0.0023, hence I am of the opinion that good American tallow, refined or not, will have an ash not very different from this per centage.

## POIVRETTE.

## CAUTION TO ANALYSTS.

BY PROF. J. CAMPBELL BROWN, D.Sc.

LET me warn members of the Society not to be misled by the presence, in some samples of pepper which are in the market, of an unusual quantity of half-bleached husk or cortical cells. They come there, either by sifting out the finer starchy portions, or by the addition of the decortications of bleached pepper or otherwise. To distinguish them from poivrette requires light not more yellow than bright daylight, and they may be mistaken during a microscopic examination by lamplight. Moreover, when the sample is boiled with dilute alkali, and washed, these cells are heavier than the dark, unbleached husk cells. The particles are of a lighter-brown colour, and have a slightly yellowish appearance to the naked eye, and they fall more rapidly than the black unbleached husk particles, much in the same way as poivrette particles similarly treated. By any one who attempts to judge merely by description, these particles might be mistaken for poivrette, especially as they are somewhat tough between the teeth. They have not, however, the same pale, bright yellow colour after alkali, nor are they of the same stony hardness as olive stones. I would also urge on the attention of every one the necessity for personally studying the several tissues from authentic samples by the aid of the notes I have given, before coming to a conclusion regarding unknown samples. No written or printed descriptions can take the place of personal study. It cannot be too strongly impressed upon the minds of students—and we are all, I hope, students—that books and papers are merely useful as guides to study, giving suggestions for, but not taking the place of, individual independent investigation.

Univ. Coll., Liverpool, 7th March, 1887.

## ESTIMATION OF MANGANESE BY PRECIPITATION WITH MERCURIC OXIDE AND BROMINE.\*

BY C. MEINEKE.

*(Continued from page 50.)*

It has already been pointed out, the precipitation of manganese is only complete from its nitric or sulphuric acid solution. From its hydrochloric acid solution, manganous oxide precipitates, which is oxidised with great difficulty by bromine. In analysing metallic iron, the best solvent is a mixture of 3 volumes of sulphuric acid of 1.13 sp. gr., and 1 volume of nitric acid of 1.4 sp. gr., adding afterwards more acid if all the iron should not be present in the ferric state. Ores of manganese, or iron ores containing manganese, are put into a small flask, and dissolved in a little hydrochloric acid. This must now be completely expelled, and the best way to do this is to introduce about 60 small glass balls, and then to boil 3 times for 10 minutes, with fresh addition of 25 c.c. nitric acid of 1.4 sp. gr. The glass balls facilitate the evaporation, and consequently the expulsion of the hydrochloric acid.

The nitric or sulphuric solution is freed from iron by precipitation with zinc oxide,

\* Repertorium der Anal. Chem.



The liquid is diluted up to a definite bulk, and an aliquot part of the filtrate, say 250 or 400 c.c., is treated as follows:—

The solution is heated to boiling, and first mixed with *precipitated* mercuric oxide, then with bromine water. If the manganese is only present in traces, no precipitate forms at first, and one, perhaps, concludes there is no manganese at all. Do not, however, neglect to add some more mercuric oxide and bromine, when, all of a sudden, even the merest trace of manganese will precipitate. If manganese is present in large quantities, the precipitate comes down at once, but it is as well to keep boiling a little longer, and to add more mercuric oxide and bromine water. If the liquid turns reddish, this is a sure sign the oxidation is complete, but if no red colour appears the rapid subsiding of the precipitate is a sign of complete precipitation. I however advise those who may try the process, to test a few c.c. of the clear liquid with nitric acid and plumbic peroxide, and should manganese still be present, a little more mercuric oxide and bromine should be added, and the boiling continued for a short time.

As a rule the sides of the beaker get thickly coated with peroxide, which is always the case if precipitation takes place before the fluid boils. If a gravimetric estimation is intended, this coating must after washing be dissolved in hydrochloric acid, and the liquid reprecipitated with bromine water and ammonia, and after washing, be ignited, together with the bulk of the precipitate. If intended for volumetric estimation, it does not matter, then it easily dissolves in oxalic acid.

After the precipitate has completely settled the liquid is if necessary decolourised with a few drops of alcohol, and poured through a filter. The precipitate is now washed with boiling water strongly acidified with nitric acid (free from nitrous compounds). Copious precipitates will now appear black, small ones will look somewhat reddish, from a mixture of mercuric oxide. The washing is continued with boiling water until all free iodine has been removed, and the filter is then at once put into a weighed crucible, and gradually heated to redness. The manganoso-manganic oxide then looks a reddish-brown porous mass.

I hoped to be able to calculate the exact amount of manganese, from the weight of the ignited precipitate, allowing for a definite quantity of co-precipitated foreign oxides. A pure manganoso-manganic oxide is certainly not always obtained. What astonished me most was to find an admixture of nickel in the analysis of metallic iron, as iron itself contains a mere fraction of this metal, as a rule. The quantity of the impurities is however generally too small to consider the direct weighing a failure. If the mercuric oxide was free from iron the impurities can only be the oxides of nickel, cobalt, and zinc. They can be speedily estimated by dissolving in a little hydrochloric acid, adding a slight excess of ammonia, and a few drops of ammoniac sulphide, finally a slight excess of nitric acid. The precipitated sulphides are then washed, and ignited when they turn to oxides. Their weight is then subtracted from the manganoso-manganic oxide. If, however, iron is present (as sometimes happens in analyses of cast-iron) this must first be precipitated by boiling the nearly neutralised solution with ammoniac acetate.

The co-precipitated oxides do not, however, interfere with the *volumetric* estimation, the principle of which has already been sufficiently explained; but there is another metal, occasionally present in ores, which interferes with the accuracy of the process, viz., *lead*.

This precipitates completely as *peroxide*, which circumstance will, I hope, enable me to work out a process for its estimation.

I now proceed to give the following test analyses, *A* being the gravimetric, *B* the volumetric results.

1. Analysis of ferromanganese (alloy of manganese and iron) containing from 81.3—81.21 per cent. of manganese.

*A.* 1 grm. in 500 c.c., used 250 c.c. = .5 grm.

Impure $Mn_3O_4$	..	..	.5683	..	..	.5680
Including NiO + ZnO	..	..	.0038	..	..	.0030

Pure $Mn_3O_4$	..	..	.5645	..	..	.5650
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=81.24 per cent. Mn =81.4 per cent. Mn.

The impure  $Mn_3O_4$  contains 99.27—99.52 per cent.  $Mn_3O_4$ .

*B.* Found 81.32—81.22 per cent. Mn.

2. Another sample of ferromanganese containing 70.28 per cent. of Mn.

*A.* 1 grm.—500 c.c., 250 c.c. = .5 grm. for precipitation.

Impure $Mn_3O_4$	..	..	.4912	..	..	.4931
Including NiO + ZnO	..	..	.0054	..	..	.0043

Pure $Mn_3O_4$	..	..	.4858	..	..	.4888
----------------	----	----	-------	----	----	-------

=69.92 per cent. Mn. 70.12 per cent. Mn.

The impure  $Mn_3O_4$  =98.88—99.11 per cent.  $Mn_3O_4$ .

*B.* Found 66.97—69.81 per cent. Mn.

3. Spiegeleisen containing 20.08 per cent. of Mn.

*A.* 2 grms. up to 500 c.c., taken 375 c.c. = 1.5 grms.; 1 grm. up to 500 c.c., taken 250 c.c. = .5 grm.

Impure $Mn_3O_4$	..	..	.4222	..	.4218	..	.1436
Including NiO + ZnO	..	..	.0050	..	.0045	..	.0032

Pure $Mn_3O_4$	..	..	.4172	..	.4173	..	.1404
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=20.01 per cent. Mn =20.05 per cent. Mn. 20.16 per cent. Mn.

The impure  $Mn_3O_4$  =98.82—98.94—98.52 per cent.  $Mn_3O_4$ .

*B.* Found 20.07—20.14—20.13 per cent. Mn.

4. Spiegeleisen containing 16.77 per cent. Mn.

*A.* 2 grms. in 500 c.c., taken 250 c.c. = 1 grm.

Impure $Mn_3O_4$	..	..	..	..	..	..	.2372
Including NiO + ZnO	..	..	..	..	..	..	.0022

Pure $Mn_3O_4$	..	..	..	..	..	..	.2350
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=16.92 per cent. Mn.

Impure  $Mn_3O_4$  =99.05 per cent. pure  $Mn_3O_4$ .

*B.* Found 16.94 per cent. Mn.

5. Ditto, containing 10.09 per cent. Mn.

*A.* 5 grms. in 500 c.c., taken 100 c.c. = 1 grm.

Impure $Mn_3O_4$	..	..	.1413	..	..	.1407
Including NiO + ZnO	..	..	.0031	..	..	.0024

..	..	..	.1382	..	..	.1383
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=9.95 per cent. Mn. =9.95 per cent. Mn.

Impure  $Mn_3O_4$  =97.87—89.29 per cent.



*B.* Found 10.01—10.01 per cent. Mn.

6. Ditto, 5 grms. in 500 c.c., 100 c.c. taken = 1 gram.

<i>A.</i> Impure $Mn_3O_4$ .. ..	·0858	..	..	·0846
Including $NiO + ZnO$ .. ..	·0021	..	..	·0017

·0837	·0829
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= 6.04 per cent. Mn      = 6.03 per cent. Mn.

*B.* Found 6.05—6.05 per cent. Mn.

7. Thomas-iron containing 3.52 per cent. Mn.

*A.* 5 grms. in 500 c.c., taken 100 c.c. = 1 gram.

Impure $Mn_3O_4$ .. ..	·0502	..	..	·0505
Including $Fe_2O_3$ .. ..	·0007	..	..	·0010
„ $NiO + ZnO$ .. ..	·0015	..	..	·0016

Pure $Mn_3O_4$ .. ..	·0480	..	..	·0479
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= 3.45 per cent. Mn.      3.46 per cent. Mn.

In impure  $Mn_3O_4$  95.59—95.14 per cent., and 1.52—1.98 per cent.  $Fe_2O_3$ .

*B.* Found 3.58—3.54 per cent. Mn.

8. Silicon iron, containing .704 per cent. Mn, 10 grms. in 1000 c.c., used 250 c.c. = 2.5 grms.

<i>A.</i> Impure $Mn_3O_4$ .. ..	..	..	..	·0274
$NiO + ZnO$ .. ..	..	..	..	·0041

·0233

= .663 per cent. Mn.

Impure  $Mn_3O_4$  = 84.12 per cent.

*B.* Found .689—.680 per cent. Mn.

9. Thomas-steel containing .603 per cent. Mn.

*A.* 9.86 grms. (instead of 10 to compensate for the volume occupied by the iron precipitate) in 500 c.c., taken 250 c.c. = 5 grms.

Impure $Mn_3O_4$ .. ..	..	..	..	·0443
Including $Fe_2O_3$ .. ..	..	..	..	·0004
„ $NiO + ZnO$ .. ..	..	..	..	·0018

Pure $Mn_3O_4$ .. ..	..	..	..	·0421
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= .606 per cent. Mn.

Impure  $Mn_3O_4$  = 95.03 per cent.  $Mn_3O_4$  and .90 per cent.  $Fe_2O_3$ .

*B.* Not carried out.

10. Crucible steel containing 1.477 per cent. Mn.

*A.* 13.66 in 1000 c.c., taken 375 c.c. = 5.05 grms. (this compensates for iron precipitate).

Impure $Mn_3O_4$ .. ..	·1082	..	..	·1087
Including $Fe_2O_3$ .. ..	·0010	..	..	·0017
„ $NiO + ZnO$ .. ..	·0039	..	..	·0035

Pure $Mn_3O_4$ .. ..	·1033	..	..	·1035
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= 1.471 per cent. Mn.      1.474 per cent. Mn.

Impure  $Mn_3O_4$  = 95.11—95.07 per cent.  $Mn_3O_4$  and .92—1.56 per cent.  $Fe_2O_3$ .

*B.* Found 1.491—1.491 per cent. Mn.

## 11. Ditto containing .841 per cent. Mn.

A. 13.33 in 1000 c.c. taken, 375 c.c. = 5.05 grms.

Impure $Mn_3O_4$	..	.0634	..	..	..	.0635
Including $Fe_2O_3$	..	.0007	..	..	..	.0008
„ NiO + ZnO	..	.0033	..	..	..	.0033

Pure $Mn_3O_4$	..	.0594	..	..	..	.0594
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= .846 per cent. Mn. .. .846 per cent. Mn.

B. Found .878—.878 per cent. Mn.

## 12. Ditto containing .427 per cent. Mn.

A. 13.33 grms. in 1000 c.c. taken, 375 c.c. = 5.05 grms.

Impure $Mn_3O_4$	..	.0312	..	..	..	.0310
Including NiO + ZnO	..	.0018	..	..	..	.0014

Pure $Mn_3O_4$	..	.0294	..	..	..	.0296
----------------	----	-------	----	----	----	-------

= .420 per cent. Mn. .423 per cent. Mn.

B. Found .421—.420 per cent. Mn.

## 13. Ditto containing .328 per cent. Mn.

A. 13.33 grms. in 1000 c.c. taken, 375 c.c. = 5.05 grms.

Impure $Mn_3O_4$	..	.0224	..	..	..	.0250
Including $Fe_2O_3$	..	.0000	..	..	..	.0010
NiO + ZnO	..	.0015	..	..	..	.0020

Pure $Mn_3O_4$ ..	..	.0209	..	..	..	.0220
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= .297 per cent. Mn. .312 per cent. Mn.

Impure  $Mn_3O_4$  = .. 93.30 .. 88.88 per cent. $Mn_3O_4$  and 0.00—4.00 per cent.  $Fe_2O_3$ .

B. Found .316—.307 per cent. Mn.

From these experiments it will be seen both the gravimetric and volumetric processes give most satisfactory results, and can be used, with any kind of metallic iron, whatever the percentage of manganese may be.

Let us now try to find out, whether there exists a constant relation between the  $Mn_3O_4$  (or  $Mn_3O_4 + Fe_2O_3$ ) and the NiO + ZnO. If we except experiment No. 8, we find the amount of NiO + ZnO is far less in the irons than in the steels. I have not separated the nickel from the zinc, but the colour of the ignited precipitate showed the former predominated.

The  $Mn_3O_4$  from the ferromanganese and cast-irons contains from 2.88-.48 per cent. of oxides of nickel and zinc. These quantities vary too much to be made the basis of a check. But in the case of steels, which contain comparatively but little manganese, it is a different thing. We can safely omit the estimation of the foreign oxides, and make an allowance instead. The iron must, however, be estimated if there is any reason to believe its presence. In the following analyses I have supposed the  $Mn_3O_4$  to contain 4.93 per cent. of zinc and nickel oxides:—



		Average of Analytical results.		Found by using the check. Percentage.	Difference.
No. 9. Thomas steel	..	603	..	600	-003
No. 10. Crucible steel	..	1479	..	1452	-022
No. 10. " "	..	"	..	1449	-030
No. 11. Crucible steel	..	850	..	859	+009
No. 11. " "	..	"	..	859	+009
No. 12. Crucible steel	..	423	..	422	-001
No. 12. " "	..	"	..	420	-003
No. 13. Crucible steel	..	315	..	303	-012
No. 13. " "	..	"	..	325	-009

In conclusion a few words about the precipitation of the manganese from a solution containing much iron.

As neither ferric sulphate nor nitrate are precipitated by mercuric oxide, it would seem, the process might be used without previous separation of the iron, and that we may simply expect a small quantity of iron in the precipitate, which at any rate would not interfere with the volumetric estimation. In trying the process with samples Nos. 3 and 5, without previous precipitation of the iron, it ended in utter failure. With samples Nos. 1 and 2 (containing but little iron) the results were, however, satisfactory.

No. 1 gave 81.46 per cent.

No. 2 „ 70.03 „

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

ON BUTTER ANALYSIS. *Zeitschr. f. Anal. Chem.*, vol. xxvi., p. 28. F.W.A. WOLL, M.Sc.—From analyses which the author has carried out and embodied in two tables, he comes to the conclusion that the determination of the melting point does not assist materially in distinguishing a natural from an artificial butter. The question can, however, as a rule, be decided by determining the sp. gravity, and by carrying out Köttstorfar's or Reichert's methods of butter analysis. He considers Reichert's method to be the one which gives the most satisfactory data for estimating the percentage of true butter in butterine.

The sp. gr. of natural butter is usually given as varying between .914 and .912; in seven samples of natural butter he obtained lower sp. gravities; the lowest being .91107.

He gives as data for natural butter—

Sp. gr. above 0.91100.

Melting point, 32–36.5°C.

Köttstorfar's method, 221.4–232.4 mgr. of potash.

Reichert's method, 12.0–14.9 c.c. of  $\frac{N}{10}$  alkali.

F.W.T.K.

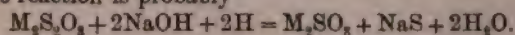
#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

QUANTITATIVE ESTIMATION OF ACIDS COMBINED WITH ALKALOIDS. P. C. PLUGGE. *Haaxman's Tydschrift*. Nov., 1886.—The acids in combination with weak opium bases, such as narcotine, papaverine, and narceine, may be volumetrically estimated with  $\frac{N}{10}$  normal soda, using either litmus, or phenol-phtalein, as indicator, as the liberated bases are too weak to affect these indicators. The amount of acid in any of the other alkaloidal salts, with the exception of coneine and nicotine, may also be titrated with soda, using phenol-phtalein. In the case of salts of codeine, brucine, morphine, or thebaine a very faint red colour makes its appearance before every trace of acid is neutralised, but this colour is readily distinguished, after some little practice, from the true red colour produced by the soda. If the salts contain free acid this must be first estimated, by using delicate litmus paper, and the phenol-phtalein is then added. Narcotine, papaverine, or narceine, must not be present. The test analyses dealing with twelve various alkaloids are satisfactory.

L. DE K.

## MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

TEST FOR HYPOSULPHITES. Prof. L. L. DE KONINCK. *Zeitschr. f. Anal. Chem.*, vol. xxvi., p. 26.—On treating the hyposulphites of the alkalis with aluminium in presence of caustic potash or soda, alkaline sulphides are formed which can then be easily detected. The reaction is probably



F.W.T.K.

ESTIMATION OF FATTY ACIDS IN SOAPS. Dr. B. SCHULZE. *Zeitschrift. f. Anal. Chem.*, vol. xxvi., p. 27.—The author gives the following slight modification of the method for estimating the fatty acids in soaps, given in Bolley's "*Handbuch der technisch-chemischen untersuchungen*," 5th edit. 1879, p. 672.

He adds dilute sulphuric acid to a weighed portion of the soap, contained in a small Erlenmeyer flask. Ether is then added, and the fatty acids which have been liberated are dissolved up in it. When the decomposition of the soap is complete, the liquid below the ethereal solution is removed by sucking it up with a pipette. With a little care this can be done very completely without any of the ethereal solution getting into the pipette. The ether solution is then shaken up with distilled water, and the latter pipetted off as before, and this process of washing repeated three times more. When all but a few drops of the wash-water have been pipetted off, the outer surface of the pipette is washed with a little ether, and a few drops of barium chloride solution added, the mixture shaken up, and the last traces of sulphuric acid thus removed. With a little practice so little water is left below the ethereal solution, that the latter can be directly poured off and evaporated. The further course of the analysis is the same as in any other fat estimation. The fatty acids obtained are perfectly free from sulphuric and hydrochloric acid, and do not get brown at 100° C.

F. W. T. K.

## APPOINTMENT.

Dr. WILLIAM JOHNSTONE has been appointed Public Analyst to the Boroughs of Dartmouth and Dunstable.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

## GERMAN v. AMERICAN FEVER.

To the Editor of the ANALYST.

SIR,—I am afraid that your learned correspondent who writes under the *nom-de-plume* of Hydroxyl is greatly mistaken in his diagnosis as to the source of that infectious malady mentioned or described by him. I can assure him that the disease is of malignant GERMAN origin, and not American; so I hope that Hydroxyl (O H)<sub>2</sub> will in future bear that in mind.

The American form of disease was thoroughly stamped out several years ago, being of a much less contagious nature than that of the Vaterland. Hydroxyl is not such an important compound radical as he supposes himself to be, and it would be exceedingly easy to "*do without him, as lots have done before*," and extremely simple to reduce his alien caustic and volatile irritability to a more oleaginous condition.—Yours truly,

ATTOLINE, Professor and Doctor.

## FOIL-LINED CAPSULES.

To the Editor of the ANALYST.

SIR,—With reference to M. Kretschmar's method of using a foil-lined capsule to obviate the difficulty sometimes experienced in (conveniently and without loss) removing a residue from the basin in which the evaporation has been conducted (abstract from *Chem. Zeitung* in the ANALYST for March), may I be allowed to say that three years ago I had some sticky extracts to work upon, which gave considerable trouble by their obstinate adhesive tendency. I then devised the plan of lining my capsules with tinfoil, and of subsequently powdering up both foil and residue together. I found this method so convenient that I have since employed it in all such cases, and can confidently recommend it to any having the above difficulty to contend with.—I am, sir, yours faithfully,

London, March 16th, 1887.

W. O. NICHOLSON.



# THE ANALYST.

MAY, 1887.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of this Society was held at Burlington House, Piccadilly, on Wednesday, the 13th ult., the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

A ballot was taken for new members and the following were declared to be duly elected :—C. E. Cassal, F.C.S., F.I.C., public analyst for Kensington ; G. H. Ogston, F.C.S., F.I.C., analytical chemist, London ; F. W. Stoddart, public analyst for Bristol ; Dr. Sedgwick Saunders, F.C.S., public analyst for the City of London.

The following were proposed for election, and will be balloted for at the next meeting :—As member, R. Davis, public analyst. As associate, Mr. Hunter, assistant to Mr. Davis.

The following papers were read and discussed :—

“On the Admixture of Starch with Yeast,” by W. F. K. Stock.

“Testing Skim Milk by the Lactocrite,” by Harald Faber.

“On another New Pepper Adulterant,” by Dr. Campbell Brown.

“On Flitwick Water,” by W. Johnstone.

The President announced that the Council had sent a subscription of £10 10s. to the Imperial Institute, on behalf of the Society.

The President also stated that another subject which interested the Society was that of pure beer ; and having regard to the deputation which recently waited on the Chancellor of the Exchequer and the views then expressed, the Council considered it would be desirable to send a circular letter to the members of the Society, drawing their attention to the employment of hop substitutes in the manufacture of beer, and requesting the members to communicate to the Secretaries any information they might have, and especially on the points whether the substances used as substitutes for hops could be detected and identified with certainty by chemical analysis, or other means, and what opinion, if any, they might have as to the effect on health of habitual small doses of such hop substitutes.

The next meeting of the Society will be held at Burlington House on Wednesday, the 11th inst.

## SOME GENERAL RESULTS OF MILK ANALYSES.

By HARALD FABER.

SOME very valuable information as to the quality of English milk in general, and the variations in the amounts of the different solids contained therein, is given in the yearly reports of milk analyses furnished by Dr. Vieth, and published in the last volumes of *THE ANALYST*. The monthly averages of the analyses given for each of the last years not only comprise a very large number of samples from over forty different farms, but they are calculated from analyses made with great care and according to a method which, in two essential points, has remained unchanged from the beginning, viz., the taking of the specific gravity, and the estimation of the total solids, these being determined by

drying 5 c.c. of milk (measured by means of correct pipettes) for three hours on an open bath, and afterwards for three hours in an air bath, the temperature of which was kept between 95° and 100° C. The full understanding of the result of this work is only with difficulty acquired in looking over so many figures; the graphical representation of the figures by means of lines on a chart will doubtless give a clearer insight by bringing the whole matter before us at a glance. Before proceeding to give such a chart I must briefly mention why I have thought it right to revise the figures given by Dr. Vieth, slightly, but, as I hope, not more than he will approve of. I treat only of the four years, from 1882-85, both included.

The specific gravity, the importance of which Dr. Vieth has so often rightly emphasized, is easily estimated with sufficient correctness with a good lactometer with an open scale. The averages for the four years were found to be 31·9, 32·3, 32·3, 32·2, viz., to vary little or nothing. The total solids, estimated as above mentioned, cannot claim for the single analysis the same accuracy as when the milk is weighed for analysis, but when we have to consider only averages of 1,000, or still more analyses, it is evident that errors in measuring must be completely eliminated, and that a fully reliable record of the total solids present in 5 c.c., understood with mathematical exactness, is obtained. The time of drying, I understand, is much longer than is the general practice, but many experiments have satisfied me that drying for a shorter period than that employed by Dr. Vieth invariably leaves some water behind, making the total solids appear too high. The estimation of fat during the first three years and the first half of the fourth was done by means of the lactobutyrometer, not, as Dr. Vieth himself informs us, because he considered it quite reliable, but because no other method practically was possible in his special work. That it did not give complete satisfaction will be evident from the following averages for the different years:—

	1882.	1883.	1884.	1885.
Specific gravity .. .. .	31·90	32·30	32·30	32·20
Total solids .. .. .	13·03	12·97	12·96	13·06
Fat .. .. .	3·52	3·50	3·74	3·93
Solids, not fat .. .. .	9·51	9·47	9·22	9·13

As the figures show, the amount of fat is gradually increasing from 3·52 to 3·93, and that of S.N.F. is decreasing from 9·51 to 9·13. And even if we exclude the last year, where another method was partly used, we find the same variation. This variation cannot be brought to harmonise with the fact that the specific gravity and the total solids remain almost constant from year to year. Fat, having a less specific gravity than water, an increase of fat must tend to lower the specific gravity of milk, and the S.N.F. giving to the watery solution a larger specific gravity than water itself, their decrease must bring the specific gravity of the milk nearer to that of water—in other words, lower it. However much anybody may disregard the specific gravity and its relation to milk solids, these facts cannot be denied; consequently, when in the above averages two causes tend to lower the specific gravity, as we go from one year to the



next, and the specific gravity still remains the same (or rather rises) something must be wrong, and in this case it is natural to consider the lactobutyrometer the source of error. Evidently it has somehow or other showed more and more of the fat, always being, according to Dr. Vieth's experience, somewhat below the truth. This, therefore, is the reason why I have considered it advisable to revise the figures, substituting for the lactobutyrometer test the method adopted by Dr. Vieth in July, 1885. I have calculated by means of Fleischmann and Morgan's formula (in "Journal f. Landw.," 1882) the amount of fat in the monthly averages from the total solids and specific gravity. Concerning the applicability of the formula I shall refer to Dr. Vieth's paper, only adding that I have tried it myself on more than 170 analyses, made with great care, spread over a long period, which convinced me of its great usefulness. The same remarks made with reference to the total solids, apply here; calculating the fat by means of the formula does not in the single analysis give very great accuracy, but the errors are eliminated when averages of such a large number of analyses are dealt with. The S.N.F. are simply taken as the differences between fat and total solids.

One thing, at least, is obtained by this revision of the figures, viz., a greater uniformity. The specific gravity and the total solids being determined during the whole time in precisely the same way, and the fat being calculated from these by means of the same formula in every case, such uniformity is insured, that whatever variations may be found from year to year or month to month may safely be attributed to differences in the milk itself, not in the analytical method. And as I purpose to show only such variations, the material, I hope, will be found sufficiently correct for my purpose.

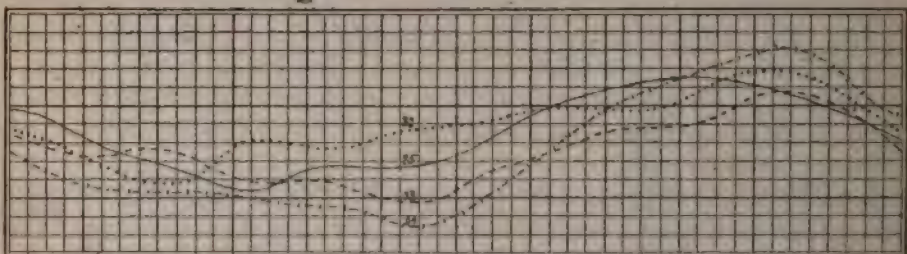
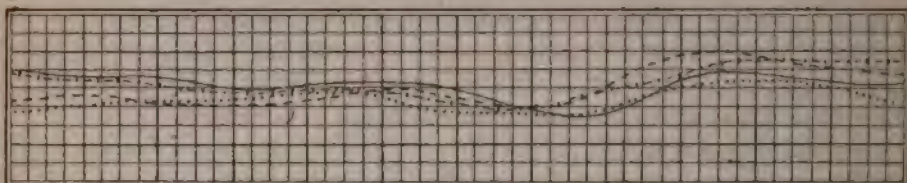
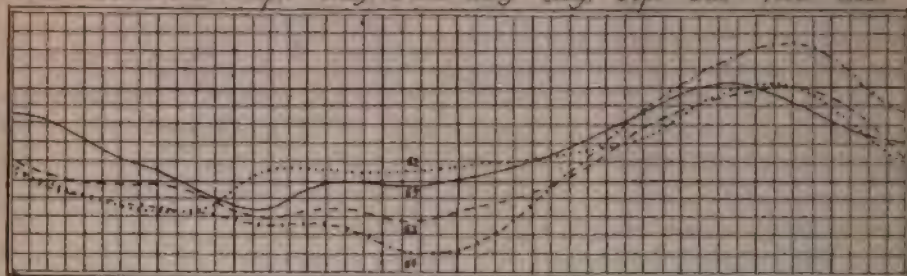
I shall now proceed to describe the chart (Pl. I.), on which is laid down by different curves the average figures for the different solids during the four years mentioned. The chart is made up of three parts; the upper part shows the amounts of total solids, the lower part those of fat, the S.N.F. being represented on the middle part. The chart is divided from left to right in twelve equal parts, one for every month of the year, as the heading shows. By horizontal lines are indicated differences in the amounts of the solids, the distance between each two lines corresponding to a difference of one-tenth per cent. Four different signatures are used for the curves of the four different years, as shown on the chart.

It will be seen immediately that there is a great difference between the three sets of curves, those for total solids and for fat presenting quite a different aspect to those for S.N.F. While in the two first-mentioned sets great differences occur from year to year and from month to month, the four curves representing the amounts of S.N.F. run very close together, and keep very nearly horizontal, the greatest difference being only 0.35 per cent. In all three sets the four curves will be found interwoven without plan, showing that while during one month the milk from one year was better, during another month it was inferior to that of the other years, but that as a whole the milk was almost alike every year, as it appears from the figures for total solids given above. But the curves for total solids and for fat, which show a great resemblance to each other (a simple consequence of the curves for S.N.F. being almost horizontal), indicate a certain variation during the year, which is found so uniformly every year that it may safely be said to be a general characteristic of English milk to vary in this manner. It

will be seen that from March to July the milk contains less solids than at other times of the year, and that from July to October a marked rise in the amount of solids takes place, the richest milk of the year being obtained during October and November.

How far this is found to be the case with milk in other countries, I am unable to state, as sufficiently correct and comprehensive analytical material is not obtainable from any other country—at least is not published, so far as I know. From some dairy trials

*Jan Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.*



..... 1882  
 - - - - - 1883  
 - - - - - 1884  
 ———— 1885

*Pl. I.*

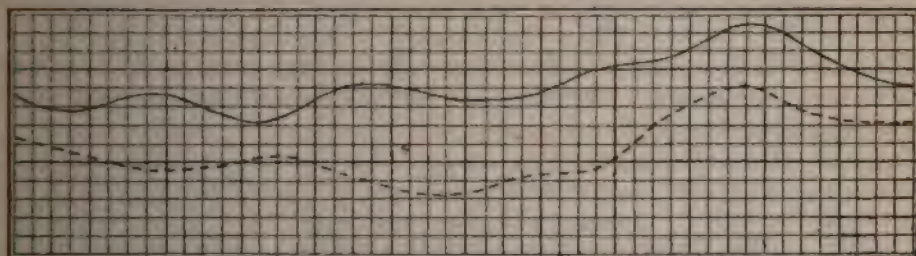
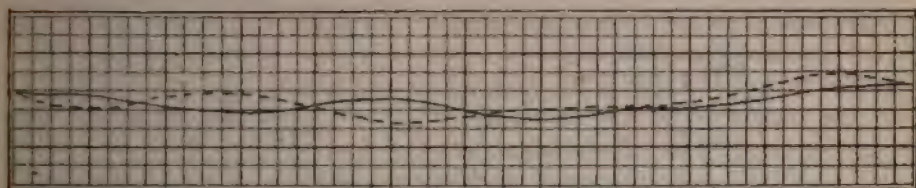
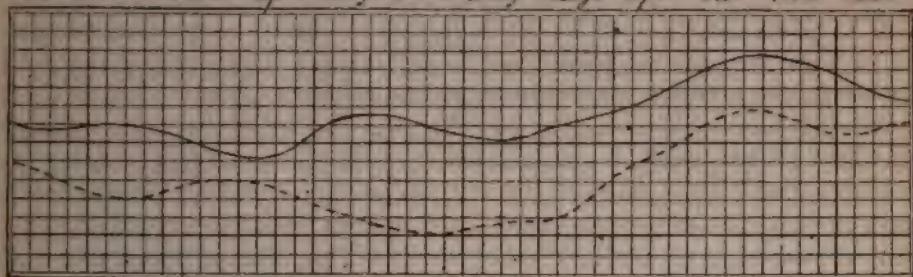
in Denmark, however, it appears likely that there also the autumn milk is the richest. This it may be of some interest to know, when discussing the cause of this variation in the quality of the milk (which discussion I shall not venture upon here), as there are some differences in the management of dairy cows in England and in Denmark. For while in England most cows calve in the spring, and consequently most of the milk in autumn and winter is milked from late-milking cows, it is the practice in Denmark to have the cows commence to come in milk very early in the autumn, in order to have plenty of milk during winter, when the price of butter is high, and the flow of milk can



be forced. This may show that the richer autumn milk cannot without further proof be explained by the fact that the cows are then getting dry. But on the other hand these curves prove how very erroneous it is to argue, as was done sometime ago in court, as a defence for a very poor sample of milk, that the milk was naturally of low quality at that time of the year (autumn) when cows were getting dry.

The results I deduce from the chart may be stated briefly as follows:—

*Jan. Febr. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.*



--- a.m. meal  
— p.m. meal.

*Pl. II.*

1. The amount of S.N.F. in average milk varies very little from year to year, and at the different times of the year.

2. The variations in the amount of total solids are for the most part due to variations in the amount of fat.

3. The amount of fat is lowest in the spring and summer, it rises from July to October, and falls again from December to January.

In order to discuss the difference between the milk from the two meals, which have not been kept separate by Dr. Vieth, I must refer to Plate II., a chart made out in

exactly the same way as the first. It gives the average results of a great number of analyses made during the year 1886 for "The Dairy Supply Company, Limited," to control the supply of milk. This firm receives a very large amount of milk drawn from about 170 farms spread over a very large part of England. The analyses are made in the following way. 5 grams. of milk are weighed, dried 1 hour on a water-bath, and then four hours in a drying oven kept exactly between  $98^{\circ}$  and  $100^{\circ}$  C. The fat is calculated from the total solids and sp. gr. by means of Fleischmann's formula with a slight modification. Uniform results may therefore be expected here also, and such as can be compared with those of Dr. Vieth, because almost the same method has been used. I have kept the two meals separate, and further information of the general character of average milk may therefore be gathered from the chart.

It will be seen that the curves for S.N.F. (in the middle part) run horizontally as in chart I., but besides it is seen, that the curve for the p.m. meal (full line) is soon a trifle above, soon below the curve for the a.m. meal (dotted line), showing that the amount of S.N.F. is the same in morning and evening milk. The variations in the amount of total solids are also here mainly due to the variations in the amounts of fat, and for both kinds of solids is the amount in the p.m. meal constantly the larger one.

The same features that are found in the other chart may be seen here, but not so clearly as the averages for both meals together are not given. To the results already given I therefore add the following:—

4. The amount of S.N.F. is the same in both meals, and

5. The amount of fat, and consequently (cfr. 2 and 4) of total solids is always larger in the p.m. meal than in the a.m. meal.

Although these results may be of greater interest to dairy-scientists than to analysts, one result which is clearly illustrated by both charts may not be without general analytical value, viz., the surprising constancy of the S.N.F. This proves how wise is the practice of basing on this item more than on anything else the decision of the character of a sample of milk. That some of the results are not quite new, I am fully aware, but to my knowledge they have not previously been borne out by a similar analytical work to that which I have been able to use here.

[NOTE.—The subject-matter of the above article was given by the author in the discussion upon Dr. Vieth's paper on the composition of milk published in our March issue. It has been since revised and submitted in its present state to Dr. Vieth, who desires to say nothing in reply, further than to emphasize the fact that during the years 1882 and 1883 the fat in the samples in question was determined exclusively by means of the lactobutyrometer. In July, 1884, this method of fat estimation was abandoned, and replaced by calculating the percentage of fat, employing Fleischmann and Morgan's formula. Since May, 1885, Fleischmann's revised formula has been used. (See THE ANALYST, vol. X., p. 69, and vol. XI., p. 68.)—EDITOR, ANALYST.]

#### ON THE ADMIXTURE OF STARCH WITH YEAST.

By W. F. K. STOCK, F.C.S., F.I.C.

*Read at the Meeting, April, 1887.*

I HAVE to confess to a considerable feeling of diffidence in bringing before this Society the subject which forms the title of my paper. As a provincial, meeting but seldom with



public analysts, it may be that I shall stand in the unfortunate position of one repeating an oft told tale. If that should unhappily be the case, please bear in mind that you owe the infliction to our honoured President, at whose invitation I stand before you.

In fairness to myself, I may say that had this request come to me two years ago I should have found it exceedingly difficult to respond, for, owing chiefly to the scattered and fragmentary nature of the literature of yeast, what I know has simply grown upon me in the pursuit of business. Looking back through twenty years of the issue of the *Chemical News* I can find but one short notice of yeast manufacture and perhaps eighteen biological references, nearly all, if not all, of which are from foreign sources.

Since it is my intention to-night to treat the addition of starch to yeast as an offence against the "Food and Drugs Act," and since my utterances will probably be made public, it will be well, at the outset, to draw particular attention to the conflicting nature of statements which have from time to time been made respecting this matter. On the one hand, I have it from manufacturers and merchants that *occasionally* starch is added to yeast to the extent of from 10 to 15 per cent. On the other hand, I have heard it declared upon oath by a member of this Society that he had analysed "thousands of samples," and had sometimes found even 75 per cent. of starch present (!) but had seldom met with pure yeast.

My own experience is that out of 120 samples recently analysed 54 contained starch varying in proportion from 5 to 40 per cent. Some samples were absolutely free from starch, whilst others contained a trace, which was proved by microscopic examination to be manufacturing residuum.

Looking at the subject from a public analyst's point of view, three questions naturally present themselves for solution. We must ask ourselves:—

- 1st. Is the addition of starch to yeast a necessity?
- 2nd. If necessary, what end does it serve?
- 3rd. What is the quantitative limit?

And these questions lead directly to a fourth, which is—Does this addition come within the meaning of the Food and Drugs Act?

I do not intend to weary you with a detailed account of how I solved these problems for myself. All I need say is that I looked in vain for help from the pages of our own excellent journal. All I met with was an exceedingly great discouragement in that the only case I could find was a prosecution for yeast adulteration, which had been quietly put to rest by words of wisdom from the magistrates' clerk, who appeared to know all about it.

However, I was fortunate in being the possessor of a translation of Pasteur's admirable "Studies on Fermentation," and by diligent reading and pretty extensive microscopic observation I arrived at a certain amount of facts which emboldened me to dare the superior knowledge of magistrates' clerks. I asked for a test case to be taken. I appeared as a witness, and a conviction was obtained. Other cases followed rapidly. In one week I travelled nearly 300 miles on yeast cases alone, and the "learned brethren" made a "rough time" for me. How it all ended is plain, when I say that the justices of the county of Durham will not now tolerate adulterated yeast.

Now let me get back to my four questions:—

Is the addition of starch to yeast a necessity? My answer to this is yes and no. Yes, if the yeast is of questionable character—that is, if it be infested with ferments other than the true torula. No, if it be an honest, healthy crop.

What end does the added starch serve? Well, in my experience, it serves the very pious end of making bad yeast look like good, and it probably impedes the movements and development of the lactic bacterium and kindred organisms. Unhappily, it does but lay them by the heels for a time. If it killed them outright, we should have cause to be thankful.

What is the quantitative limit? I feel unwilling to answer this question, for, after hearing sworn testimony to the finding of 75 per cent., it might prove erroneous to put it anywhere within 100 per cent.

The fourth question, as to the capacity of the Act to deal with this fraud, is squarely answered by a long and growing list of convictions in my own county.

Before proceeding to speak of the methods I have adopted for the analysis of starched yeast, permit me, for a moment, to deal with the social bearings of my subject. There are here involved questions of health which are in importance far superior to that of the petty fraud committed in the substitution of cheap starch for the more expensive yeast. So far as my experience goes, starch has the effect of making yeast presentable to the eye when it is absolutely unfit for use. Good bread cannot be made from bad yeast, and whatever deteriorates the staple food of a people deteriorates also the social condition of that people.

I come now to the analysis of yeast under the "Food and Drugs Act, 1875." I think I am safe in saying that methods hitherto published are not entirely satisfactory, but in presence of a company of trained analysts I am clearly open to correction. But I believe I may say, without fear of contradiction, that a trustworthy, *direct* method is still a desideratum among analysts.

In my own practice I have met with only four or five varieties of starch in yeast, which I now mention to you in the order of the frequency of their occurrence, namely:—Potato starch, maize starch, rice starch, arrowroot and buckwheat starch. The two last named are rare, and, indeed, I have some doubt about the buckwheat. It is difficult to differentiate this starch from rice starch.

Perhaps the simplest method that ever suggested itself for the separation of starch from yeast is that of deposition from a dilute aqueous diffusion, and if carried out in a special manner, it leaves, with potato and arrowroot starches, nothing to be desired. But with maize starch, rice starch, or buckwheat starch, it is quite impracticable, owing to the fact that the rates of deposition of these three are more or less coincident with the deposition rate of yeast itself. Numerous experiments have shown this to be beyond question.

The matter stands quite differently with potato starch and arrowroot, and since potato starch is at present the commonest adulterant of yeast, I have been led to adopt the following method for its determination. The sample is broken down and well mixed; 25 grammes are weighed on a pair of fine scales; the weighed portion is transferred to a glazed mortar, and gently rubbed down with 50 c.c. of cold water to a smooth cream, then washed carefully into a *clean* 30 oz. beaker. Next 5 grammes of pure potato farina are treated in exactly the same way, and transferred to a second



clean 30 oz. beaker. The contents of both beakers are now diluted rapidly, with a strong stream of water from the town supply (which is made to issue through a  $\frac{1}{4}$ -inch glass nozzle attached by I-R. tubing to the tap) until the column of liquid in each is four or five inches in height, which height must be constant for both, and is best marked for guidance. The beakers are now allowed to stand at perfect rest until the one containing the P. starch only has suffered complete deposition. Both are now carefully decanted, and the water quickly rushed up again to the mark. This is repeated until the rate of deposition for each is alike when it is only necessary to decant the perfectly clean water from the beaker which contained the sample, wash out the starch on to a filter, allow to drain, wash twice with strong alcohol, once with ether, and dry gradually, finishing at 100° C. When dry the starch is swept off the filter paper into a counterpoised watch-glass, and weighed. Yeast mixed with arrowroot is dealt with exactly as above, with the precaution only of using arrowroot starch for a standard of deposition. After weighing, the only further step to be taken in the case of unmixed starch residues is to correct for loss on drying at 100° C. I have found, by two concordant experiments, that air dried potato starch loses 16.50 per cent. of its weight, when heated for two hours in the water-bath, and two concordant experiments gave 15.04 per cent. loss for arrowroot. If it be found that the dried residues are contaminated with matters not starch, such as malt dust, rye awns, or mineral matters, the necessary corrections must be made. The *modus operandi* of these corrections is obvious.

I have here the details of two experiments made by this method with mixtures of pure yeast, and potato starch and arrowroot respectively in known proportion.

	Potato Starch.	Arrowroot.
Grammes of yeast taken .. ..	20.00	20.00
„ starch .. ..	5.00	5.00
Percentage of starch .. ..	<u>20.00</u>	<u>20.00</u>
	Potato Starch.	Arrowroot.
Grammes of dry starch recovered ..	4.126	4.181
Add, for loss in drying at 100° C. ..	.815	.737
Grammes of air dried starch recovered	<u>4.941</u>	<u>4.918</u>
Percentage of starch found in mixture	19.764	19.672
Percentage of total starch recovered	98.82	98.36

As before stated, maize, rice, or buckwheat starches are not amenable to treatment by deposition. After many trials I have been compelled to resort to micro-methods for their quantitative estimation. A preliminary micro-examination having indicated the presence of one of these starches I proceed as follows:—

Five grammes of the sample are weighed out, brought to a smooth paste in a glazed mortar with the addition of a little water. The mixture is now gradually diluted until it measures 200 c.c. A drop of the well-stirred mixture is transferred by aid of a glass rod to a micro-slide having a glass slip cemented along one edge. A minute drop of solution of iodine in aqueous—not alcoholic—solution of potassic iodide is next added,

and the whole is now evenly mixed with the rod by strokes alternating at right angles, taking care not to carry the liquid beyond the limits of the cover glass, which I prefer of an inch square in size. The cover glass is now placed in position, and lifted rapidly four or five times with a mounted needle, after which the slide is examined with a quarter inch object, and B eyepiece giving 360 or so diams. The iodised granules stand out in fine relief, and in the case of maize are easily counted. Rice is more difficult to manage owing to the greater number of granules, but I have had a special eyepiece prepared, which has two fine lines crossing at right angles in the centre of the field-lens, thus dividing the field into four equal segments. This little modification has been a great comfort in counting. This counting I perform over ten distinct fields, and thus get a fair average. I then repeat the whole of these very simple operations with a mixture of genuine yeast, and the same variety of starch, which starch I add to the extent of 20 per cent. It is only necessary to do this once for all, if fixed conditions as to weight of sample, bulk of liquid, size of rod, and area of cover glass, along with degree of amplification, are observed. The standard once established serves for all proportions. I state this as the result of experiment, in proof of which take the following:—Two mixtures of rice starch with yeast were made; one contained 20 per cent. starch; the other was made with yeast, already known to contain about  $1\frac{1}{2}$  per cent. of starch, to which 10 per cent. further was added. On counting by the foregoing method, 1502 granules were found in ten fields of the 20 per cent. mixture, and 844 granules for ten fields of the  $11\frac{1}{2}$  per cent. mixture—giving in round numbers an average respectively of 150 and 84 per field. Then if  $150 = 20$  per cent.,  $84 = 11\frac{1}{2}$  per cent.—a result which, considering all things, is sufficiently gratifying.

There is just one other point I should like to touch upon in conclusion, and I mention it to put analysts upon their guard in cross-examination. I have found sometimes that samples of yeast taken from the same consignment, and out of the same batch, will vary in their content of starch by perhaps 5 per cent., giving rise apparently to serious discrepancies in analyses. This occurs chiefly with potato starch, and it is due to the fact that in the manufacture of mixed yeast, the starch and yeast are pumped into the filter press simultaneously from the mixing box, and, as we have seen, the starch deposits, and so the tail end of the batch is apt to get an accumulated dose of starch.

#### DISCUSSION.

The PRESIDENT said this was just an instance of the advantages afforded by the Society. They here had a member who had had special experience in his district of an article which had been neglected in others, and it was very important that his special experience should be laid before the Society, in the form of a paper, as had been done. He would ask any other gentleman who had had any experience with it to express his opinion on the plan adopted by Mr. Stock, and to make any comments on the amount of starch, if any, which could be tolerated.

Mr. HEHNER said: I would like to have seen some information as to the reasons which induce Mr. Stock to say that there is no use or necessity for the presence of starch in yeast, and why he asserts it to be only required in yeast of bad quality. I may state that my experience is diametrically opposed to that of Mr. Stock. Some



years ago I made many practical experiments with yeast, and found that with some yeasts, and those the most active and powerful, it was impossible to bake a good loaf of bread, until a large proportion of starch had been added, with a view to dilute the yeast and diminish its activity. Indeed the better the yeast and the quicker its action, the greater was found the necessity of the addition of a neutral body. The bakers are accustomed to add a certain quantity of yeast to a given amount of flour, and they cannot be induced to diminish the quantity of yeast in proportion to its activity; it is not reasonable to expect them to have an analysis of the yeast before their mind's eye. With yeast we expect to get a certain effect; the effect only is aimed at; I should say, if a sample containing 90 per cent. of starch did its work properly and well, it should be permitted in preference to a pure but weak article. I would of course oppose any deception in this matter, but analysts, as such, are not, in my opinion, in a position to judge of the requirements of the baker. The activity of a yeast in breadmaking depends greatly upon the temperature at which the yeast has been cultivated. As these temperatures vary in the case of brewers or distillers' yeast, or that specially bred for the purpose, the activity must be controlled by the addition of judicious quantities of starch.

Mr. HARVEY said that in Canterbury yeast was sent out perfectly pure, and with no starch added. He apprehended that the consistency of it had something to do with what Mr. Hehner had said. The yeast goes out in a massive state, requiring a knife to cut it. He was not aware where it went to, nor whether bakers did anything with it.

Mr. STOCK, in reply, said that Mr. Hehner had really touched the gist of the whole matter when he said that brewers' yeast could not be used without some intermediate cultivation, but he thought Mr. Hehner had missed the object of his paper.

If any analyst were not to oppose this sort of thing he did not quite see where they were getting to. Mr. Hehner had stated that if 90 per cent. of starch were necessary it was a fair thing to add that quantity, but he differed from Mr. Hehner.

Some which he examined contained no starch whatever, but the question was how were they to prevent the unnecessary addition of starch, unless the Sale of Food Act could take knowledge of it?

There was another point from the bacteriological point of view. In samples of yeast containing starch that merchants had sent him he found that the bacteria had multiplied by thousands, and the yeast was absolutely unfit for use. How far was that sort of thing to be allowed to go on?

Of course he did not say that starch was injurious in yeast.

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#### ON ANOTHER NEW PEPPER ADULTERANT.

By PROF. J. CAMPBELL BROWN, D.Sc.

*Read at the Meeting, April, 1887.*

In the *Produce Market Review*, for February 26th, 1887, vol. xxii, No. 1044, p. 103, mention is made of Dhari as being used for admixture with pepper. This probably means Dhoura—Dhari being the flower of a plant, or probably several plants used in dyeing—known also in Southern India as Cholum, and in Bengal as Jowari. I have met

with it only about four times in pepper, but it probably occurs more frequently in other districts. It is known in England as Great Millet, or Turkish Millet, and is the grain (with an integument, but without the husk) of one of the cereal grasses, *Sorghum Vulgare*.

It is a roundish or oval somewhat flattened grain, size from  $\frac{1}{8}$ th to  $\frac{1}{4}$ th of an inch in diameter, white in colour and brittle, with a thin smooth integument, or testa, showing under a high microscopic power, on the inner surface, an aggregation of very small granules, which become blue by iodine. The body of the seed is very white, and consists mainly of roundish or irregular starch granules, varying in size from '0001 up to '0006 of an inch in diameter, and showing under polarised light a nearly right angled cross; and of larger irregularly rounded granules of starch from '0005 up to '0013 of an inch in diameter, showing no cross, or only a very faint one, under polarised light.

Some of the first named granules have a hilum and star in the centre, somewhat like bean starch. By boiling with caustic alkali, the cellular membrane which binds the starch granules together is disclosed.

The influence of an admixture of Sorghum with pepper upon analysis of the latter will be seen from the following analysis of Sorghum grains:—

Moisture 11 per cent.

Composition of the dried sample.

	I.	II.
Ash .. .. .	1.31	1.69
Soluble in 10 per cent. hydrochloric acid ..	90.70	87.80
Starch .. .. .	75.20	73.00
Albuminous matters soluble in caustic alkali	6.71	7.96
Cellulose .. .. .	2.56	4.19
Alcoholic extract .. .. .	10.36	7.96
Ethereal extract .. .. .	10.10	7.30
Nitrogen .. .. .	1.82	1.79

A sample of ground Dhoura is submitted to the Society with this paper.

University College, Liverpool.

## FLITWICK WATER.

By WILLIAM JOHNSTONE.

(Read at the Meeting, April, 1887.)

THIS singular ferruginous water wells up through a bed of irony looking peat and decayed vegetable matter, being in a valley cut down, through the Lower Greensands, to the underlying Oxford clay.

The locality is known as Flitwick Moor, and at one time served to supply the inhabitants of the district with fuel. The Flitwick Station of the Midland Railway is within a mile of the springs, and about forty miles from London.

The Flit, a tributary of the Ivel, runs in the valley, taking its rise some distance westward, near the junction of the Greensands with the overhanging Gault Clay. At Flitwick the valley opens out, and there is a considerable spread of flat land, rich in



alluvial deposits. These succeed each other at different levels above the present bed of the stream—marking the different heights at which, in past times, the river flowed—a river, probably, but feebly represented by the Flit. The peat occupies a middle place, between the highest terraces of gravel and the modern alluvium, a narrow strip of clay fringing both sides of the water.

Several attempts have been made to drain this valley, but have been abandoned, owing to insufficiency of fall, so that, at the present time, much of the place remains in its original wild state. Morass and heathery waste revel together in uncultivated luxuriousness—happy hunting ground for rare and curious water-loving plants and insects. The surrounding country is mostly sand; for the most part irony, loose, and incoherent, but sometimes rocky. There is much poor ironstone, as concretionary lumps and nodules\*—the whole mass being deeply stained with iron peroxide, which has produced a varied assemblage of coloured sands, according to the different stages of oxidation. The floor of the valley supporting the sands being Oxford clay, and impermeable to water, I should conclude that the phenomenon of the springs is the joint production of the rainfall percolating the sands being stopped by the clay, and that percolating the porous irony peat being likewise stopped by the clay. There is no reason to suppose the water must rise from any depth below, fissures, or the like.

A peculiarity that may be noticed is that the irony water coming to the surface, and running in open channels, deposits its iron; so that a tufaceous looking deposit of bog iron ore is being formed around—whereas, where the water preserved for use runs into covered troughs, the iron is held in suspension. An enormous quantity of available Flitwick mineral water may thus be seen meandering to waste amongst the woods and bogs that mark the place.

#### ANALYSIS OF THE WATER COLLECTED IN BEGINNING OF MAY, 1885.

	Parts per 1,000.	Grains per Imp. Gallon.
$(\text{NH}_4)_2\text{O}$	·1144	8·008
CaO	·0640	4·480
MgO	·0511	3·577
$\text{Fe}_2\text{O}_3$	1·2212	85·484
$\text{Al}_2\text{O}_3$	·0044	0·308
CuO	·0075	0·525
MnO	·0014	0·098
$\text{K}_2\text{O}$	·0024	0·168
Na	·0211	1·477
Cl	·0340	2·380
$\text{SO}_3$	1·7328	121·296
$\text{SiO}_2$	·2328	16·296
Organic matter	1·2753	89·271
	<hr/> 4·7624	<hr/> 333·368

\* See *Geol. Magazine*, Aug. 1886, p. 381.

## SEVERAL CONSTITUENTS IN COMBINATION.

	Parts per 1,000.	Grains per Imp. Gallon.
$(\text{NH}_4)_2\text{SO}_4$	·29040	20·3280
$\text{CaSO}_4$	·15525	10·8675
$\text{MgSO}_4$	·15340	10·7380
$\text{Fe}_2\text{SO}_4$	1·92138	134·4966
$\text{Al}_2\text{SO}_4$	·01470	1·0290
$\text{CuSO}_4$	·01508	1·0556
$\text{MnSO}_4$	·00299	0·2093
$\text{K}_2\text{SO}_4$	·00527	0·3890
$\text{NaCl}$	·05520	3·8640
Iron Apocrenic	1·39000	97·3000
Iron Crenic	·26840	18·7880
$\text{SiO}_2$	·23280	16·2960
Organic Matter	·25730	18·0110
	4·76217	333·3519
Total by Direct Estima- tion at 140° C. . .	4·8000	336·000
Specific Gravity at 15·5° C. 1·0041.		

One peculiarity of this water is its freedom from alumina, seeing that it contains such a large quantity of iron, and that in this instance wholly as ferric salt, containing, as you will observe, 1·92 grm. of ferric sulphate per 1,000, and 1·65 grm. of organic iron salt. The water, however, is not quite constant in composition, varying, to some extent, with the rainfall containing at times ferrous as well as ferric salts; that, however, I believe, is due to the more rapid extraction of the iron salts from the peat by excessive heavy rain, and, therefore, not allowing time for complete oxidation.\*

As to its medicinal properties, I leave those to my medical friends, merely drawing your attention to the following analysis of the water, the origin of which has been reputed to two individuals (see ANALYST, Vol. X., p. 29, and *British Medical Journal*, 3rd Jan., 1880). Whoever is responsible for the analysis in question is sadly deficient in his knowledge of even the elementary rudiments of chemical combination. Just fancy, Mr. President, a water containing 144 grains of oxide and carbonate of iron, as represented in the following analysis; they are practically insoluble.

	Grains per Gallon.
Oxide and Carbonate of Iron .. .. .	144·00
Sulphate of Magnesia and Soda .. .. .	59·20
Carbonate of Lime .. .. .	11·54
Muriate of Magnesia (Chloride) .. .. .	15·16
Silica .. .. .	3·40
Carbonate of Magnesia .. .. .	5·42
" Soda .. .. .	0·22
Ulmic, Malic, and other Acids, constituents of Vegetable Matter .. .. .	13·32
Total .. .. .	252·26

\* A sample collected on 22nd March last year contained an abundance of both ferric and ferrous salts, and also gave 473·90 grains of solid residue per imperial gallon.



I must admit that I was not personally aware of the existence of *malic acid* in peat, or decomposed vegetable matter such as peat.

In conclusion I have to thank my friend, Mr. A. C. G. Cameron, of H.M. Geological Survey, for the interesting description of the locality of the spring.

*Conclusion of the Society's Proceedings.*

#### A METHOD FOR THE SEPARATION AND ESTIMATION OF BORIC ACID.

*With an Account of a Convenient Form of Apparatus for Quantitative Distillations.*

By F. A. Gooch.\*

In all successful methods for the estimation of boric acid, its comparative isolation is a necessary preliminary. Fortunately, the removal of nearly everything which interferes seriously with the proper execution of methods is not particularly arduous; but, of ordinarily occurring substances, two, silica and alumina—both very commonly associated with boric acid—are especially annoying in this regard. In the separation of alumina the trouble lies in the tendency of the precipitated hydrate to carry and retain boric acid,† so that the two cannot be parted by means of ammonia or ammonia salts; with silica, the difficulty is in removing it completely. The volatility of boric acid stands, of course, absolutely in the way of treating with acid and evaporating to dryness, and every chemist knows the vainness of attempting to precipitate silica by means of ammonia, ammonia salts, or zinc oxide in ammonia. In Stromeyer's method‡ the presence of silica is peculiarly harmful, since in passing to the condition of potassium fluosilicate this substance nearly quadruples its weight, and to free the potassium fluoroborate from contaminating fluosilicate requires, according to Fresenius,|| at least six treatments by solution in boiling water, the addition of ammonia, and evaporation to dryness. Wöhler§ recommends evaporating the hydrochloric acid solution to dryness in a flask fitted to a condenser, collecting the distillate, reuniting the latter with the residue, and filtering from silica; and the operation is successful so far as the complete removal of silica is concerned; but the alumina, if present, is still in condition to give annoyance, and the other bases are yet to be separated.

Advantage has long been taken of the volatility of free boric acid with hydrofluoric acid or with alcohol to secure its removal from fixed substances, but so far as I know no attempt has been made heretofore to secure its complete volatilisation and estimation in the distillate. The experiments which I proceed to describe are the result of an effort to accomplish this end.

Aside from the difficulties in manipulation, and in the construction of apparatus which the use of hydrofluoric acid would involve, this reagent is otherwise plainly inapplicable to the purpose in view, and of other agents with which boric acid is known to volatilise freely methylalcohol seems to present the most desirable qualities. Methylalcohol, ethylalcohol, and water are effective in the order in which they are named.

\* American Chemical Journal.

† Wöhler, Ann. d. Chem. u. Pharm. 141, 268.

‡ Ann. d. Chem. u. Pharm. 100, 82.

|| Quant. Chem. Anal. 424.

§ Handbook of Mineral Analysis, under "Datholite."

Thus, to volatilise 1 gram. of boric acid—the equivalent, speaking roughly, of about 0.5 gram. of boric anhydride—two treatments with 10 c.c. of methylalcohol and evaporation to dryness in each case were adequate; for the volatilisation of 0.2 gram. of boric acid were required two treatments of 10 c.c. each of ethylalcohol, succeeding an evaporation with 50 c.c. of the same alcohol; and the residue of five evaporations of water over 0.4 gram. of boric acid, taking in each case 50 c.c. of water, followed by ignition, weighed 0.08 gram. or one-fifth of the original weight. In the presence of water, methylalcohol is not equally effective; amylalcohol and sulphuric acid restrain its action similarly, doubtless by dilution simply, and hydrochloric acid seems to possess no advantage over water alone in developing the volatility of boric acid. As an example, an experiment may serve in which a solution of 0.4 gram. of boric acid in 50 c.c. of water, after being heated three times successively with 25 c.c. of methylalcohol until the boiling-point rose in every case nearly to that of water, and then evaporated to dryness, left a large residue which disappeared with a single charge of 25 c.c. of methylalcohol applied by itself.

From the residue of the evaporation of borax with hydrochloric, nitric, or acetic acid, methylalcohol, as would naturally be predicted, volatilises the boric acid freely, though the presence of foreign material acts to a certain degree protectively, and tends to diminish the rapidity with which the alcohol would otherwise effect extraction and volatilisation. In case, however, that acetic acid is used to break up the borate, the tendency of sodic acetate to lose acid and become alkaline simply by exposure to evaporation in its aqueous solution makes it necessary to insure the acidity of the residue of evaporation by adding a drop or two of acetic acid before repeating the treatment with methylalcohol.

On the whole, methylalcohol shows itself to be an excellent agent by which to secure the volatilisation of boric acid.

To retain free boric acid, magnesium oxide naturally suggests itself. According to Marignac,\* it is effective, and if, in the course of analysis, it may have been partly converted to the chloride, it is easily regenerated by the action of heat and moisture. Marignac, it will be remembered, makes use of magnesia mixture—the chlorides of ammonium and magnesium with free ammonia—to fix the boric acid, evaporating the solution to dryness, igniting, extracting with boiling water, filtering, and weighing the residue, while the filtrate is again treated as before to recover traces of the borate, which has yielded to the solvent action of the water. During the drying and ignition the magnesium chloride yields hydrochloric acid, and it would seem scarcely possible that the magnesium borate should fail to show some loss of boric acid when both hydrochloric acid and moisture exert their action. Further, the presence of ammonia during evaporation does not prevent the volatilisation of boric acid, and Marignac regards the addition of it from time to time as of doubtful use. So it appears natural to look for some loss under such conditions, and Marignac fully recognises the fact that the apparent accuracy of his method is due to the balancing of errors, the inclusion of foreign matter by the magnesium borate, and the deficiency of the magnesia when precipitated as ammonio-magnesium phosphate together compensating for the loss of boric acid by volatilisation.

*(To be continued in our next, with Illustration.)*

\* Zeit. für Anal. Chem. 1, 406.



## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

THE LIMIT OF FUSEL OIL IN SPIRITS. G. BODLANDER and J. TRAUBE. Repert. Anal. Chemie., No. XI. A question much discussed now-a-days is, What percentage of fusel oil ought to be allowed in brandies and spirits? Many prosecutions of distillers have either been withdrawn or been dismissed, as no fixed standard had been agreed upon by analysts. The only chemist who has proposed a standard is Dr. Beer, whose opinion has been often quoted in court. He gives .3 per cent. as the highest limit. One of the authors has already pronounced his own opinion in a previous paper, and thinks this figure is much too high, as the results of analyses of a great number of samples of good and inferior corn brandies, by means of Traube's capillarmeter (which gives results within .05 per cent.), showed the amount of fusel oil to seldom exceed .2 per cent.

The best way out of the difficulty would have been to make physiological experiments, but the authors finally decided to settle the question by a great number of experiments with the cheaper kinds of brandy.

Whatever may be the physiological effect of fusel oil, most hygienists will agree, the less a sample of spirits contains of it the better, and the public has a right to insist upon a thorough purification even of the cheaper kinds of spirits.

The results of ten investigations of cheap corn brandies by means of the capillary process are given. 1. Description of sample. 2. Percentage by volume of alcohol. 3. The specific gravity of the sample at 15° C after having been diluted to about .976 sp. gr. 4. Temperature of laboratory at the time of observation. 5. The height of column in millimetres. 6. The corrected height at 15° C and .976 sp. gr. 7. The amount of fusel oil in *diluted* sample. 8. Percentage in original sample. (A pure spirit of wine of 20 per cent. gave following results: 3 = 796, 4 = 15, 5 = 52.3, 6 = 52.3, 7 = 0, 8 = 0.)

1	2	3	4	5	6	7	8
A	34	.9759	14.5	51.25—51.25	51.25	.12	.20
B	35.2	.9759	14.5	50.90—50.90	50.90	.17	.30
C	37	.9758	14.5	51.40—51.40	51.50	.10	.19
D	32.5	.9757	15.0	51.55—51.70	51.80	.06	.10
E	36	.9761	15.0	51.60—51.70	51.60	.09	.16
F	34	.9762	14.8	52.00—51.90	51.80	.06	.10
G	34.7	.9760	14.8	50.80—50.65	50.70	.19	.33
H	34.5	.9761	15.0	51.05—51.10	51.05	.15	.26
J	34.5	.9758	15.0	51.30—51.50	51.50	.10	.17
K	36.3	.9764	15.0	51.60—51.60	51.30	.12	.22

From this table it will be seen only *one* brandy exceeded Dr. Beer's limit, and this was not one of the *cheapest* samples. The authors therefore think the limit ought to be put lower, say .15 per cent.—L. DE K.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

ESTIMATION OF ALKALOIDS IN EXTRACTS. BY E. DIETERICH. Pharm. Central, 1887.—(a) Examination of Extracts of Belladonna, Aconite, Conium, and Hyoscyamus.—Triturate 0.2 grms. of powdered lime, prepared from marble, with 3 grms. of water; add

2 grms. of extract ; when dissolved carefully add 10 grms. powdered lime. The mixture is then placed in a closed continuous displacement apparatus, the receiving bottle containing about 30 grms. of ether is suspended over a water bath (not too hot), and the process of extraction is regulated by bringing the ether bottle nearer or further from the water bath. With extracts of belladonna, aconite, and hyoscyamus, extraction is carried on for thirty or forty-five minutes at the highest. Conium extract requires at least two hours. It is advisable to exhaust a second time with ether. The ethereal solution of the alkaloids is transferred to a tared porcelain capsule, and the receiving bottle rinsed two or three times with small portions of ether ; 1 c.c. of distilled water is added, and the ether carefully evaporated over a water bath at a temperature not exceeding 30°C., care being taken not to work near hydrochloric, nitric, acetic, or other volatile acid. The residue, weighing 1.5 grms., is dissolved in 0.5 c.c. of alcohol, specific gravity .892, the solution diluted with 10 c.c. distilled water, and, after adding one or two drops of rosolic acid solution (1 : 100 alcohol), titrated with 100th normal sulphuric acid, each cubic centimetre of which neutralises 0.00289 grms. atropine or hyoscyamine, 0.00523 grms. aconitine, and 0.00127 conine.

The following results were obtained :—

*Extract of Belladonna* (13 experiments) : 1.170, 1.184, 1.163, 1.170, 1.156, 1.142, 1.156, 1.142, 1.156, 1.170, 1.184, 1.170, 1.170 per cent.

*Extract of Aconite Root* (6 experiments) : 1.305, 1.252, 1.279, 1.252, 1.279, 1.279 per cent.

*Extract of Hyoscyamus* (6 experiments) : .780, .766, .766, .751, .751, .766 per cent.

*Extract of Conium* (6 experiments) : .609, .597, .622, .622, .597, .589 per cent.

(b) *Examination of Extract of Nux Vomica*.—0.2 grms. powdered lime and 1 gm. extract of nux vomica are intimately mixed, 3 c.c. of distilled water added and evenly mixed with 10 grms. powdered lime, then exhausted in the same manner as before, for one and a half or one and three quarter hours. The receiving bottle is rinsed with alcohol twice and then with ether, and after adding 1 c.c. of distilled water the percolate is evaporated in a tared porcelain capsule (at the same temperature and with the same caution as stated above) to 1.5 grms. ; then add 0.5 c.c. alcohol, specific gravity .892, 10 c.c. of distilled water and two drops of rosolic acid solution, and titrate with 1.20th normal sulphuric acid. Toward the end it is advisable to use delicate blue litmus paper conveying the solution on the paper by means of platinum wire ; 1 c.c. of 1.20th normal sulphuric acid corresponds to 0.0182 grms. of alkaloid.

The method may be modified by triturating 1 gm. extr. nux vomica with 3 c.c. normal ammonia, and adding 10 grms. powdered lime.

The first process yielded the following results of six experiments : 18.74, 18.92, 18.74, 18.56, 18.65 per cent. alkaloid.

W. H. D.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ANALYSIS OF CLAY. MEINEKE. *Rept. Anal. Chemie*, 14.—No analysis of clay is trustworthy unless the silica has been examined for alumina, and the alumina for silica ; then it must not be supposed the errors compensate one another. To obtain



accurate results the author proceeds as follows: .5 gramme of the clay is mixed in an agate mortar with a few grammes of sodic bicarbonate and then heated to intense redness in a platinum dish. The fused mass is treated with boiling water, decomposed with excess of hydrochloric acid, and then evaporated to dryness in the water bath. To completely render the silicic acid insoluble, the author heats in the air bath for some time to 150° C. A higher temperature, though unnecessary, is not hurtful, as the drying may even be done at 300° C. After cooling, the mass is drenched with fuming hydrochloric acid, allowed to stand for half an hour, and treated with cold water. The insoluble silica is washed five times by decantation, and finally washed on the filter until free from chlorine, when it is at once transferred to a weighed platinum crucible, and gradually heated to redness. If the mass does not readily get white this is usually caused through imperfect washing. After weighing the silica it must be digested in hydrofluoric acid and the liquid evaporated to dryness. Any alumina or ferric oxide must now be weighed and subtracted from the weight of the silica.

If the filtrate from the silica is boiled in glass or porcelain vessels, even of the best kind, a not inconsiderable amount of silica dissolves, and is afterwards precipitated with the alumina. The author, therefore, again evaporates to dryness in a platinum dish, when, on treating with dilute hydrochloric acid, a further small quantity of silica is obtained. If, however, the alumina has also to be estimated, the liquid is not evaporated, but any silica afterwards separated from the weighed alumina, by digesting in dilute sulphuric acid (1-1).

The usual plan for estimating the amount of sand in clay is to heat with sulphuric acid, then to boil with water, and to collect insoluble residue on a weighed filter, or, better still, a tared filter. After weighing, an aliquot part is boiled with strong solution of sodic carbonate, which dissolves the silica and leaves the sand insoluble. As, however, the use of weighed filters is inconvenient, the author adopts the following plan: The residue, after treating with sulphuric acid, is put on a filter and, after washing, dried for some time until it seems perfectly dry all through. The filter is now exposed for some time to the air until the weight is constant. The greater part of the precipitate can now be easily removed from the filter, only a small quantity, sometimes no more than .03 gram, remaining. The bulk of the insoluble matter is now weighed, and then divided in two equal parts. Part *a* is ignited, to get moisture; part *b* is boiled with sodic carbonate, to get the sand. The filter is burned, and a small quantity *c* mixture of anhydrous silica and sand is obtained, in which the proportion of sand can then be easily calculated.

L. DE K.

#### CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

To the Editor of the ANALYST.

SIR,—The letter in your March number, signed "Hydroxyl" opens out a question bearing closely upon the fair fame of our little Society of Public Analysts, which the majority of our members would, I believe, jealously guard. Though small and somewhat young, the Society has, by dint of sound, active work—in which it will yield the palm to no other—and honourable, straightforward conduct, hitherto held a position of which its members are justly proud, and which they will expect the Council to uphold by denouncing, and if possible preventing, any insidious, unprofessional conduct on behalf of its members, such as that your correspondent so justly denounces.

A correspondent, grandiloquently subscribing himself "Attoline, Professor and Doctor," has replied to the letter in your current issue so petulantly that, in vulgar parlance, he is plainly a person whom



"the cap fits," and being so he would have shown more discretion if he had somewhat bated his spleen; he should have recollected that "those who live in glass houses cannot throw stones."

I know not who he may be, but he evidently opines that "Hydroxyl" is "OH," in whom your readers will recognise one of our two able and courteous secretaries. The Society's success has been largely due to its having always been fortunate in securing presidents and secretaries who have been devotedly attached to it and have been unremitting in freely bestowing upon it the full force of their energies and abilities. These disinterested self-sacrificing qualities are possessed by our present office-bearers in a degree which has not been excelled by any of their predecessors.

Your correspondent was not wise in his impertinent and uncalled-for sneer about "Hydroxyl's" alien origin, for if I am correct in my surmise, though he is more English than many natives of our land, and is sincerely attached to the country of his adoption, which is benefited by his choice, yet he is proud to have belonged to that great German nation which has given to the world perhaps a larger proportion of masterly chemists than any other. How offensive and presumptuous that sneer becomes when we call to mind that several of our most highly esteemed members hailing from that part of the world have done some of the best work amongst our number, and that for years past they, with a sprinkling of our own countrymen, have, by their activity and perseverance, been the mainstay of our Society. I enter my protest against that sneer, which applies not alone to "Hydroxyl" but equally to all those other honourable and highly gifted members who are of the same nationality, and I take this earliest opportunity of saying that I feel assured it will be indignantly repudiated by the Society. "Attoline's" other sneer, that "it would be exceedingly easy to 'do without him, as lots have done before'" is singularly unhappy, seeing that he shows us in his letter he does not even know how to write the formula for hydroxyl. Rather than scoff at "Hydroxyl" let him go to him and take advantage of his help, so good-naturedly and ungrudgingly bestowed upon all members of our Society who ask it, and let him there learn to write (OH.)

This hankering after empty titles is not confined to any one profession. I recently saw a learned professor described in a placard of a course of lectures as "F.R.S., F.G.S., F.R.G.S., F.L.S., F.Z.S." How ridiculous! Such caudal appendices always give me the impression of belonging to *quadrumanous* rather than to *bimanous* mammalia. Should it not be kept in mind that it is not the qualification which makes the man, but that it is the man who makes the qualification, and that "bogus" degrees are not only contemptible and derogatory to their possessors, but reflect in a most prejudicial manner on a Society, when it has become contaminated with them.

Is it not evident that when "Hydroxyl" wrote as he did he was fearful lest the good name of our Society, whose interests he has worked so untiringly to promote, should be sullied by the recent ill-advised action of some of its members? I, for one, think that the warning has been sounded not a moment too soon, and fully endorse his timely suggestion that those members who are the pitiable sufferers from the infectious disorder he has so accurately diagnosed and so faithfully portrayed should be subjected to control, lest they should contaminate others. Would that they could be dealt with after the methods prescribed by the various Contagious Diseases (Animals) Acts, and Orders in Council made thereunder! I am sure we may rely upon the Council of our Society to guard its honour by doing whatever lies within their power to prevent it from being decimated by the virulent infectious disease so reasonably dreaded by "Hydroxyl."—I am, Sir, yours faithfully,

ALFRED ASHBY.

Grantham, April, 1887.

#### DETECTION OF HOP SUBSTITUTES IN BEER.

To the Editor of the ANALYST.

SIR,—In view of the circular just issued to the members of the Society of Public Analysts, and of the contributions invited on the detection of hop substitutes, the following references to articles on the subject, published in English periodicals, etc., may not be without interest to your readers:—

Systematic method of examining beer for bitter principles. Dragendorff. Jour. Chem. Soc., xxvii. (1874), 818.

Improved scheme for detecting chief bitter principles. Dragendorff. Jour. Chem. Soc., xlii., 103; Year-Book Pharm., 1882, 120.

Useful scheme for detecting chief hop substitutes. Wittstein. Jour. Chem. Soc., xxix., 767.

Tabular scheme for separating hop-bitter, and detecting chief hop substitutes, chiefly based on a method described by Enders. Allen. Commercial Organic Analysis, i., 97.

Method of detecting aloes, colocynth, wormwood, gentian, larch-fungus, scammony and jalap. Bach. Jour. Chem. Soc., xxvii., 923; or far more perfectly abstracted in Year-Book Pharm., 1874, 293.

Detection of Aloes in beer. Bornträger. Year-Book Pharm., 1880, 140.

Presence of a natural substance in beer, giving reactions simulating certain of those produced by colchicine, gentian-bitter, etc. Jour. Chem. Soc., xxxi., 326; xxxii., 809.

Detection of noxious bitters in beer. Dupré. ANALYST, viii., 40.

Reactions of picrotoxin. Year-Book Pharm., 1872, 251; 1881, 48; Chem. News, xxxix, 264; xlvii., 249. Jour. Chem. Soc., xl. 286, 440; xlii., 412; xlviii., 449; l., 284.

Isolation and characters of quassia. Christensen. Jour. Chem. Soc., xlii., 1,302; Pharm. Jour., (3) xlii., 146. Adrian and Moreaux, Jour. Chem. Soc., xli., 908; Pharm. Jour. (3), xiv., 507. Oliveri and Denaro, Jour. Chem. Soc., xli., 1192; Pharm. Jour. (3), xv., 409.

The references to the detection of picric acid in beer would themselves form a considerable list. The method described by me (Com. Org. Analysis, i, 98) leaves nothing to be desired in this direction.

—I am, Sir, yours truly,

Sheffield, April 23rd, 1887.

ALFRED H. ALLEN.



# THE ANALYST.

JUNE, 1887.

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*(The Journal is this month enlarged, and entirely devoted (by special order of the Council) to reporting the Proceedings at the Special Meeting on the Beer and Butterine Bills.)*

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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

### SPECIAL REPORT OF A MEETING TO DISCUSS THE BILLS RELATING TO THE PURITY OF FOOD NOW BEFORE PARLIAMENT.

A meeting of this Society was held at Burlington House, Piccadilly, on Wednesday, the 11th ult., the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened it was announced that the following gentlemen had been elected: As members:—R. H. Davies, public analyst; Bertram Blount, analyst. As associate:—T. Hunter, assistant to Mr. Davies.

The following gentlemen were proposed for election, and will be balloted for at the next meeting:—W. T. MacAdam, public analyst for Portobello; T. W. Glass, assistant to Dr. Redwood.

The President opened the meeting by calling attention to the bills now before the House of Commons, affecting the food supply of the Country. These bills are as follows:—

### A BILL FOR BETTER SECURING THE PURITY OF BEER (A.D. 1887).

WHEREAS it is expedient, with a view to enable the public to distinguish between beer brewed from hops and malt from barley and beer composed of other ingredients, to amend the law relating to the sale of beer:

Be it therefore enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:

[Short title.] 1. This Act may be cited as the Pure Beer Act, 1887.

[Declaration of ingredients on selling beer.] 2. Every person who sells or exposes for sale, by wholesale or retail, any beer brewed from or containing any ingredients other than hops or malt from barley shall keep conspicuously posted at the bar or other place where such beer is sold or exposed for sale a legible notice stating what other ingredients are contained in such beer. Any person who sells or exposes for sale any such beer as aforesaid without complying with the above enactment shall be liable to a fine not exceeding in the case of the first offence *five pounds*, and in the case of the second or any subsequent offence *twenty pounds*. Any fine incurred under this section may be recovered summarily by any informer, and one half of the fine shall in every case be paid to the informer.

[*Definition of Beer.*] 3. In this Act the term "beer" includes beer (other than black or spruce beer), ale, and porter.

[*Commencement of Act.*] 4. This Act shall come into operation on the *first day of January one thousand eight hundred and eight-eight.*

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A BILL FOR BETTER SECURING THE PURITY OF BEER (A.D. 1887).

WHEREAS it is expedient, with a view to the better protection of the public from adulteration of beer, to amend the law relating to the sale of beer :

Be it therefore enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows :

[*Short title.*] 1. This Act may be cited as the Beer Adulteration Act, 1887.

[*Penalty on selling beer containing other ingredients than hops and malt without giving notice.*] 2. Every person who sells or exposes for sale by wholesale or retail any beer brewed from or containing any ingredients other than hops and malt from barley, shall keep conspicuously posted at the bar or other place where such beer is sold or exposed for sale a legible notice stating that other ingredients are contained in such beer.

Any person who sells or exposes for sale any such beer as aforesaid, without complying with the above enactment, shall be liable to a fine not exceeding in the case of the first offence *forty shillings*, and in the case of the second or any subsequent offence *ten pounds*.

Any fine incurred under this section may be recovered summarily by any informer, and one half of the fine shall in every case be paid to the informer.

[*Definition of Beer.*] 3. In this Act the term "beer" includes beer (other than black or spruce beer), ale, and porter.

[*Extent of Act.*] 4. This Act shall not extend to Ireland.

[*Commencement of Act.*] 5. This Act shall come into operation on the *first day of January one thousand eight hundred and eighty-eight.*

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A BILL TO REGULATE THE IMPORTATION, MANUFACTURE, AND SALE OF BUTTER SUBSTITUTES. (A.D. 1887.)

BE it enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons in this present Parliament assembled, and by the authority of the same, as follows :

[*Short title.*] 1. This Act may be cited as the Butter Substitutes Act, 1887.

[*Definitions.*] 2. In this Act the word "butter" shall mean such article produced from unadulterated milk or cream unmixed with any other fatty or oleaginous substance whatsoever.

The words "margarine" or "oleomargarine" shall mean any imitation of butter whatsoever, or any compound of butter with animal fat, or any compound of butter and animal or vegetable oil.

[*Must brand all packages distinctly.*] 3. From and after the passing of this Act any person or persons importing, manufacturing, or offering for sale any margarine or oleo-



margarine shall cause to be stamped or branded distinctly and durably on every tub, firkin, box, or case containing the same intended for sale the word "margarine" or "oleomargarine" in letters not less than one inch in length.

[*Penalty.*] Any person or persons acting in contravention of the provisions of this section shall, on summary conviction, be each and severally liable for the first offence to a fine not exceeding *twenty pounds*, for the second offence to *one month's* imprisonment or a fine of *fifty pounds*, and for any subsequent offence to *six months'* imprisonment.

[*Manufactories to be registered.*] 4. Every manufactory of "margarine" or "oleomargarine" within the United Kingdom of Great Britain and Ireland shall be registered with the clerk of the union in which such manufactory is situated, and shall be open at all reasonable hours to inspection by the inspectors appointed under the Sale of Food and Drugs Act, 1875.

[*Penalty.*] Any person or persons carrying on the manufacture of margarine or oleomargarine without having caused the premises in which the same is carried on to be registered, in compliance with the provisions of this section, shall be liable, on summary conviction, to a penalty not exceeding *five pounds* for every day in which such manufacture is carried on without the registration hereby directed.

[*Invoicing.*] 5. Every manufacturer of and every wholesale dealer in margarine or oleomargarine shall clearly state on every invoice of such article that the goods therein invoiced are sold as margarine or oleomargarine.

[*Penalty.*] Any person or persons who shall neglect to comply with the provisions of this section shall, on summary conviction, be liable to a fine of *one hundred pounds*, and shall not be entitled to recover from the purchaser the value of goods not invoiced as herein directed.

[*Retail dealers shall inform purchasers. Penalty.*] 6. Every retail dealer in margarine or oleomargarine shall in every case inform the purchaser of any quantity of the same that the article sold is margarine or oleomargarine, and every retail dealer acting in contravention of the provisions of this section shall, on summary conviction, be liable, for the first offence, to a fine of *ten pounds*, for the second offence to a fine of *twenty pounds*, and for the third or any subsequent offence to *one month's* imprisonment without the option of a fine.

[*Reward for information securing conviction.*] 7. Any person giving such information as shall lead to a conviction under this Act shall be entitled to and receive half the amount of the fine imposed.

[*Proceedings same as under Sale of Food and Drugs Act, 1875.*] 8. All proceedings under this Act shall be the same as prescribed by sections twenty, twenty-one, twenty-two, twenty-three, twenty-four, twenty-five, twenty-six, twenty-seven, twenty-eight of the Sale of Food and Drugs Act, 1875, and all officers employed under that Act are hereby empowered and required to carry out the provisions of this Act.

[*Regulations for describing correctly on forwarding by public carrying companies.*] 9. Any person or persons forwarding any consignment of margarine or oleomargarine by steamship, railway, canal, or other public conveyance shall state clearly on the bill of lading, way-bill, or other consignment ticket the true description of such goods as herein-before mentioned, and every steamship, railway, canal, or other public carrying company

shall enter the same on their books, manifestoes, and other entries or document under that description.

[*Penalty.*] Any person or persons acting in contravention of this section shall, on summary conviction, be liable to a penalty of *ten shillings* for each package on every consignment not booked as required by the provisions of this section.

[*Goods in transitu may be examined.*] 10. Custom House and Inland Revenue officers shall be entitled to examine goods *in transitu* for the purpose of ascertaining that the provisions of this Act are not violated, and to employ experts, if necessary, in cases of urgency.

#### A BILL FOR THE BETTER PREVENTION OF THE FRAUDULENT SALE OF OLEOMARGARINE. (A.D. 1887.)

WHEREAS it is expedient that further provision should be made for protecting the public against the sale as butter of oleomargarine or other substances made in imitation of butter, as well as of butter mixed with any such substances.

Be it therefore enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons in this present Parliament assembled, and by the authority of the same, as follows :

[*Short title.*] 1. This Act may be cited as the Oleomargarine Act, 1887.

[*Definition.*] 2. The word "butter" shall mean the substance usually known as butter, made exclusively from milk or cream, or both, with common salt, and with or without additional colouring matter.

The word "oleomargarine" shall mean all substances, whether compounds or otherwise, prepared in imitation of butter, and whether mixed with butter or not, and no such substance shall be lawfully sold, except under the name of oleomargarine, and under the conditions set forth in this Act.

[*Penalty.*] 3. Every person dealing in oleomargarine, whether wholesale or retail, whether a manufacturer, importer, or as consignor or consignee, or as commission agent or otherwise, who is found guilty of an offence under this Act, shall be liable on summary conviction for the first offence to a fine not exceeding *twenty pounds*, and for the second or any subsequent offence to a fine not exceeding *fifty pounds*.

[*Marking of cases.*] 4. Every person dealing in oleomargarine shall conform to the following regulations :

Every barrel, tub, packet, or case, whether open or closed, and containing oleomargarine, shall be marked "Oleomargarine" on the top, bottom, and sides, in large, printed capital letters, and if such oleomargarine be exposed for sale in any open plate or vessel, there shall be attached to such open plate or vessel, so as to be clearly visible to the purchaser, a label marked in large printed capital letters "Oleomargarine."

And every person selling the same shall in every case inform the purchaser at the time of sale that the substance sold is not butter.

[*Presumption against vendor.*] 5. Every person dealing with, selling, or exposing, or offering for sale, or having in his possession for the purpose of sale, any quantity of oleomargarine contrary to the provisions of this Act, shall be liable to conviction for an offence against this Act, unless he shows to the satisfaction of the court before whom



he is charged that he purchased the article in question as butter, and with a written warranty to that effect, that he had no reason to believe at the time when he sold it that the article was other than butter, and that he sold it in the same state as when he purchased it, and in such case he shall be discharged from the prosecution, but shall be liable to pay the costs incurred by the prosecutor unless he shall have given due notice to him that he will rely upon the above defence.

[*Reward for information.*] 6. Any person giving such information as shall lead to a conviction under this Act shall be entitled to and receive half the amount of the fine imposed.

[*Oleomargarine imported or manufactured.*] 7. All oleomargarine imported into the United Kingdom of Great Britain and Ireland shall be duly entered as such by the officers of Her Majesty's Customs, and all oleomargarine, whether imported or manufactured within the United Kingdom of Great Britain and Ireland, shall, whenever forwarded by any public conveyance, be duly consigned as oleomargarine; and it shall be lawful for any officer of Her Majesty's Customs or Inland Revenue, if he shall have reason to believe that the provisions of this Act are infringed on this behalf, to detain and examine any barrel, tub, packet, or case, and ascertain, if necessary by analysis, whether an offence against this Act has been committed.

[*Registration of manufactory.*] 8. Every manufactory of oleomargarine within the United Kingdom of Great Britain and Ireland shall be registered from time to time in such manner as the Local Government Boards of England and Ireland respectively may direct, and every person carrying on such manufacture without being duly registered shall be guilty of an offence under this Act.

[*Proceedings.*] 9. All proceedings under this Act shall be the same as prescribed by sections twelve to twenty-eight inclusive of the Sale of Food and Drugs Act, 1875, and all officers employed under that Act are hereby empowered and required to carry out the provisions of this Act.

In reply to the circular letter on the subject of "Beer," which had been sent out by instructions of the Council, Mr. Hehner read the following communications, viz.:—

Mr. F. M. RIMMINGTON writes:—In my public capacity of analyst I have had to examine many samples of beer, and must confess that it is most difficult to arrive at any satisfactory conclusion respecting the nature and character of the bitter principles employed.

In the first place the amount of any active principle contained in the substance used as a hop-substitute will be extremely minute in a pint of the malt liquor; so that unless a large quantity is operated on nothing tangible is obtained. When hops are used there is no difficulty in recognising the presence; but if substitutes are used it is an impossibility with our present knowledge to isolate and identify the bitter principles of such agents as gentian, chiretta, quassia, calumba, etc. The process I generally adopt is to evaporate a measured portion of the sample to the consistency of an extract, and then to digest this with alcohol, and evaporate the solution to dryness, and treat the residue with ether, chloroform, or amylic alcohol—this residue I generally examine microscopically.

Respecting the question of healthfulness of such substances as I have named above, as substitutes for the hop in beer, I am quite of opinion that they are as wholesome, if not more so, for such a purpose, as the hop itself, which is known to possess narcotic properties—of course I limit this opinion to the substances named; and very good beer is brewed without hops.

Dr. STEVENSON writes :—With reference to the Council's questions (1) and (2), I have to remark that, in my opinion (1) the leading substances used, or alleged to be used, in brewing as substitutes for hops cannot be detected and identified with such certainty as is demanded in a court of law by chemical analysis alone. They may, however, be detected and identified with certainty when such analysis is supplemented by physiological experiments. There are some substances thus used, or alleged to be used, which cannot, I believe, at present be detected with certainty by these means conjointly.

(2) I know of no evidence to show that the effect on health of the habitual use of small doses of such hop-substitutes as are now used is injurious. That they are injurious when taken in much larger doses than could be well introduced into beer, and that they are injurious to the lower animals, does not prove them to be injurious in habitual and minute doses to man. We need good observations and experiments in this direction.

A difficulty is created by "The Cruelty to Animals Act, 1876," commonly known as the Vivisection Act. It is an error to suppose that this Act merely prohibits—except under license—operations on living animals. The best legal authorities tell me that the administration of any substance to an animal, if the administration is calculated to give pain, is penal, provided the experiment be made by way of experiment. Any administration of hop-substitutes to any vertebrate animal with the view of testing the effects of the substance is likely to bring the experimenter under the lash of the law. And even a licensed person cannot test the quality of a beer by such experiments, for the Act restricts the experiments to the advancement of physiological knowledge, the saving or prolonging of life, and the alleviation of suffering. No one, I believe, has power under the existing statute to grant a license or permission to experiment on animals in the carrying out of the Sale of Food and Drugs Act.

I think this word of warning is necessary, as I know of instances where public and other analysts have unwittingly infringed the law in the discharge of their duties.

Mr. A. W. SMITH, Rye, Sussex, writes :—In the first place I am of opinion that very little of so-called hop-substitutes is used in the southern counties, the probable substitutions being more to economise in malt than in hops. The price of hops during the past few years has been abnormally low, and therefore no advantage can be gained by using anything else. Again there is no other vegetable bitter with which I am acquainted so pleasant to the taste, nor so grateful and soft to the palate, as the hop, nor is there one, that I am aware of, which will keep so well.

As the chairman of a local committee, appointed to inquire into and report upon the "Depression of Trade" for the Royal Commission, the hop question came before me, as it is upon the cultivation and price of hops that we in Sussex so much depend, and I naturally feel some interest in the matter. I have, therefore, taken the opportunity—whenever it has offered—of endeavouring to ascertain whether substitutes were really much in use, and if so, what they were composed of. The only sample I have seen consisted mainly of quassia-wood, with a small portion of gentian and valerian roots finely ground and mixed.

There is no doubt that saccharine substances of various kinds (sugar, glucose, and an article called saccharine, etc.) are used in making cheap beers, but these of course do not furnish the bitter principle. I fear also that various articles, used ostensibly as "finings," are not altogether free from objectionable characteristics, such, for instance, as tartaric and salicylic acids, both of which in some persons cause uneasiness in the stomach and bowels. Some beers again appear to have diuretic and aperient properties, but I have never attempted to ascertain the cause; probably carbonic acid gas would be responsible for the latter.



If any bitter other than hops is used, I have no doubt it would be either quassia, gentian, chiretta, or calumba (or perhaps a mixture of them), but I think these (the latter excepted) can easily be detected by Tr. Ferri Perchlor. I append a table showing the results of a few experiments in this direction. Bisulphide of lime is extensively used for scouring and cleansing vats and vessels, and it is more than probable that some of it finds its way into the lower grade of beers as a preservative agent.

But I happen to know for a fact that brewers of fine ales not only use hops exclusively, but obtain the very best samples our country produces, and one gentleman I know, who is a grower, and grows good East Sussex hops too, and many more than his brewery requires, yet buys still better hops for his choicest ales. There are, however, people still insane enough to believe that strychnine is yet used by some.

When searching for salicylic acid, it is well to heat the sample under examination in a test tube, so as to cause some froth at the top, the violet colour showing much more definitely in the white beads.

TABLE SHOWING RESULTS OF SAMPLES OF ALES TREATED WITH THIS REAGENT (TR. FERRI PERCHLOR) :—

No. 1. Ale, malt, and hops only.	No. 2. Malt, hops, and quassia.	No. 3. Malt and quassia.	No. 4. Malt, hops, and gentian.
Dark grey discolouration and cloudiness, with deposit.	Grayish discolouration and cloudiness.	Colour intensified—cloudiness, and grey flocculent deposit.	Blackish discolouration, much cloudiness, and deposit.
No. 5. Malt, hops, and chiretta.	No. 6. Malt and hop-substitutes.	No. 7. Malt and calumba.	No. 8. Malt, hops, and salicylic acid.
Black discolouration, much cloudiness, and dark deposit.	Very decided dark, discolouration and cloudiness, with black deposit.	No particular change, but normal colour slightly intensified.	Violet discolouration, cloudiness, and deposit.

Dr. SEATON read the following notes upon recorded observations respecting the physiological effects of hops, quassia, gentian, chiretta, and chamomile :—

All the above-mentioned bitters are used for medicinal purposes, and are contained in the British Pharmacopœia.

The infusion and tincture of hops are mild but agreeable aromatic tonics. They sometimes prove diuretic, or, when the skin is kept warm, sudorific. They are said to be narcotic, but the existence of narcotic qualities has been strongly denied by Bigsby, Majendie, and others. Majendie tried the different preparations of hops on animals, and did not observe any narcotic properties, although, as he observes, "the narcotic property is one which is most strikingly displayed in experiments on animals."

Gentian in full doses has a laxative effect upon the bowels, and may produce vomiting. It is doubtful whether it possesses narcotic effects. Majendie swallowed two grains of gentianin dissolved in alcohol, but only observed extreme bitterness and a slight feeling of heat in the stomach. But Planche has shown that the distilled water of

gentian causes violent nausea, and within three minutes a kind of intoxication. Buchner, in his toxicology, has a record of a narcotic effect produced in Prussia by the medicinal use of the drug, although the presence of foreign matter could not be detected. But the deleterious effects said to have resulted from the use of gentian may have been due to a foreign root intermixed with it.

Chiretta possesses the same qualities as gentian in its tendency to relax the bowels. There is no record of its possessing any narcotic qualities.

Quassia, says Pereira in his *Materia Medica*, in the usual medicinal doses is a stomachic and tonic—that is, it is bitter to the taste, promotes the appetite, and assists the digestive functions. It is devoid of all irritant stimulant and astringent properties, and has been, therefore, sometimes taken as a type of the simple or pure bitters. It is more powerful than, but in other respects analogous to, gentian in its operation. “We can find nothing in this wood,” says Dr. Cullen, “but a pure and simple bitter.” There is evidence to show that it has a narcotic effect on animals.

Does it act as a narcotic on man as on other animals? Pereira says, “I have employed, and seen others administer, quassia most extensively, but never had grounds for suspecting any effect of the kind alluded to.”

Quassia, like other bitters, checks putrefaction, and is said to be superior to other bitters in this respect.

Chamomile infusion has an effect upon the alimentary canal. Thus, given in large doses, it is sometimes used to assist the action of emetics. It is also, according to Ringer, useful in checking the summer diarrhoea of children in the early stage.

It appears, therefore, that the bitters—quassia, gentian, chiretta, and chamomile (which are all in the pharmacopœia)—possess some of the qualities of hops, but are less decidedly medicinal. Quassia is not diuretic, and has neither laxative nor astringent action on the bowels; and there is no evidence of its being narcotic.

Gentian and chiretta are only slightly laxative, and there is no evidence of their being narcotic.

Chamomile, in certain conditions of the mucous membrane of the bowels, has a slight astringent effect, but is otherwise innocuous.

Mr. C. HEISCH says:—With respect to bitters those which are *known* to be injurious we can deal with at present; those which are most commonly employed, such as gentian, calumba, chamomile, and chiretta, there is every reason to believe are at least as wholesome as hops. Moreover, I know of no way of speaking with certainty as to either of them in the quantities of beer which we could work on, or in the time we could give to the examination.

It seems to me that the use of bad glucose, containing large quantities of chlorides, often purposely produced by acting on starch with hydrochloric acid, and simply neutralising with either potash or soda, and boiling down, is much more of a fraud than the general run of hop-substitutes; though Mr. Bannister, in his remarks at the Society of Arts, speaks of ales containing 120 grs. of chlorides derived either from the water or malt adjuncts as of no consequence. I believe a return to the old rule of not permitting more chlorine than is equivalent to fifty grs. chloride of sodium would be a good thing, would stop the use of much bad water in brewing, and also the use of such glucose as mentioned above; but I see no reason to stop the use of sugar or other wholesome malt adjuncts properly prepared.

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The following papers were then read and discussed:—



## AN IMPROVED METHOD OF DETECTING QUASSIA AND CERTAIN OTHER HOP-SUBSTITUTES IN BEER.

BY ALFRED H. ALLEN.

*Read at the Meeting, May, 1887.*

HITHERTO, the detection of hop-substitutes in beer has had for English analysts little more than an academic interest. There has been no definition of beer, nor standard of strength or quality, and hence the brewer has been free to employ any hop-substitute that could fairly be regarded as non-injurious. Now, however, that there are two Bills before Parliament, both of which aim at rendering the employment of hop-substitutes illegal, unless duly acknowledged, the question has acquired considerable practical importance. If the Bills in question ever become law, it will devolve on the public analyst to certify to the presence or absence of hop-substitutes, but, as was recently pointed out by the Chancellor of the Exchequer to a deputation which waited on him, it would be of no use to pass an Act forbidding the unacknowledged use of hop-substitutes, unless it was possible to detect infringements of the law. Hence it has become important for public analysts to see how far the more important hop-substitutes can be definitely detected in beer, or at any rate distinguished from hops.

The problem in question is by no means a simple one. Beer itself is a highly complex and variable product, and some of the normal constituents add to the difficulty of detecting hop-substitutes. The problem is further complicated by the possible presence of several substitutes simultaneously, together with actual hops. Then the bitter principles, to which the hop-substitutes owe their employment, have in some cases been very imperfectly studied, and belong to a class of bodies by no means distinguished for strong chemical affinities or for characteristic reactions. In fact, the most general and striking property of the majority of hop-substitutes is the intensity of their bitter taste, a character which materially increases the difficulty of detecting them owing to the very moderate amount employed to give the beer the desired flavour. There is one other complication of practical importance, and that is the very considerable quantity of beer usually recommended to be used for the analysis. Thus if two litres be used for the main examination, as is recommended by Dragendorff, at least twice that quantity should be submitted to the analyst, and hence twelve litres would have to be purchased. The necessity of purchasing so large a quantity as two and a half gallons of each beer would almost certainly render the Act abortive.

In looking into the question, therefore, I have aimed at reducing the amount of beer employed as low as possible.

In the last number of the ANALYST I gave a list of references to articles in English periodicals, etc., connected with detection of hop-substitutes in beer. These I have carefully studied, and they have formed the foundation of the following statements and proposals.

Dragendorff, who has published a very elaborate method for the recognition of a large number of possible hop-substitutes (*Jour. Chem. Soc.* xxvii, 818 ; xlii, 103), operates on two litres of beer, precipitates the concentrated liquid with basic acetate of lead, further concentrates the filtrate and adds a large volume (1000 to 1200 c.c.) of absolute alcohol! The spirit is subsequently driven off, and the various principles extracted by a systematic employment of immiscible solvents.

Wittstein (Jour. Chem. Soc. xxix, 767) operates in a similar manner, but works on one litre, and omits the treatment with acetate of lead, as indeed did Dragendorff in his older method. In the method described by me in my "Commercial Organic Analysis" (vol. i., page 97), based on a process of Enders, one litre of beer is employed, the concentrated liquid treated with alcohol, the filtrate precipitated with ether, and the filtered liquid evaporated, the residue redissolved in alcohol, treated with water, and the solution precipitated with acetate of lead.

Several of the writers on the subject state that on precipitating a beer with basic acetate of lead the hop-bitter is wholly precipitated, and hence, if the concentrated filtrate still have a bitter taste, the presence of some hop-substitute is certain.\* This difference seemed to me so important that I have very carefully investigated it, and find it perfectly in accordance with the fact. I prefer, however, to employ *neutral* acetate of lead instead of the basic or ammoniacal acetate, as the latter reagents are liable to precipitate certain bitter principles not removed by the first. On the other hand, the hop-bitter is very perfectly precipitated by neutral lead acetate, and this statement is equally true of an infusion of hops purposely prepared and of a beer in which hops is the sole bitter used; but I have some reason to think that basic acetate of lead is liable to effect a less perfect separation of the hop-bitter.

In attempting to improve these processes, I have endeavoured to dispense with the use of alcohol, and yet separate the bitter principles from the sugar and other bodies which disguise the bitter taste and interfere with application of chemical tests, and find that a very satisfactory product for further treatment can be obtained by the following simple method.

One litre of the beer is evaporated to about 300 c.c., and is then precipitated while hot with a solution of neutral acetate of lead. The precipitate is filtered off, the filtrate allowed to become cool, and any further precipitate is also removed. The excess of lead is then removed from the filtrate by sulphuretted hydrogen, and the liquid filtered and further concentrated to about 150 c.c. I prefer sulphuretted hydrogen to sulphuric acid or a sulphate, as the lead sulphide seems to carry down a notable quantity of colouring matter.

The treatment with lead acetate removes all tannin, phosphates, etc., and *the hop-resin and lupulin are also completely precipitated, while all or nearly all hop-substitutes remain in solution.*

The next object is to separate the sugar, dextrin, and mineral constituents of the beer as perfectly as possible from the active principles of the various hop-substitutes. Instead of precipitating the carbo-hydrates, etc., by excess of strong alcohol, I prefer to remove the bitters from the aqueous liquid itself by agitation with suitable immiscible solvents.

The object being to extract as many active principles as possible in the simplest possible way, leaving their mutual separation and recognition for further consideration.

\* This distinction between the bitter principles of hops and hop-substitutes is referred to in the work entitled "*Chemistry applied to the Arts and Manufactures*," edited by Chas. Vincent, and often described as the "New Edition of Muspratt's Chemistry." The process is also described in Wynter Blyth's work on the "*Analysis of Food*," and forms an essential part of Enders' method of detecting bitter substances, so that there is a very general consensus of opinion as to the value of the test.



I employ chloroform, as having the most general solvent action. It separates with tolerable ease from the aqueous liquid, and should be employed as long as it leaves a notably bitter residue on evaporation. In many of my experiments I relied on the traces of lactic and other acids naturally present in beer to produce the requisite degree of acidity, but I am now of opinion that the addition of a little dilute sulphuric acid is advantageous, if not actually necessary, in some cases. The extraction with chloroform being complete, ether should next be used, the treatment being repeated as long as any notably bitter principle is extracted. Finally, the aqueous liquid is rendered alkaline with ammonia, and agitated with chloroform or ether-chloroform, to extract any alkaloids.

The following arrangement shows the behaviour of the more important bitter principles when the aqueous liquid is agitated in succession with chloroform, ether, and ammonia and ether-chloroform. I have personally verified the behaviour of the substances to the names of which asterisks are attached.

1. Extracted by chloroform from acid solutions :—

- Absinthin (wormwood).
- \*Anthemini (chamomiles).
- Colchicine (colchicum), imperfectly.
- \*Colocynthin (colocynth, or bitter apple), imperfectly.
- \*Calumbin, and probably some berberine (calumba), bright yellow, and highly fluorescent.
- \*Gentipierin (gentian), very imperfectly.
- \*Picric acid (artificial), yellow, imperfectly.
- Picrotoxin (*cocculus indicus*), with difficulty.
- \*Quassiin (quassia wood).

2. Subsequently extracted by ether from acid solutions :—

- \*Chiratin (chiretta).
- \*Colocynthin (colocynth, or bitter apple).
- \*Gentipierin (gentian).
- \*Picric acid, yellow.
- \*Picrotoxin (*cocculus indicus*).

3. Subsequently extracted by ether-chloroform from alkaline solutions :—

- \*Berberine (calumba root).
- Colchicine (colchicum).

By evaporating off the solvent, warming the residue with a little alcohol, and then adding water, solutions are obtained, which will be bitter if any of the above substances be present. A very small quantity of the substance is required for this test; indeed the use of too large an amount must be carefully avoided, or the sense of taste will be found to be wholly paralysed, for the remainder of the day at any rate.

It will be seen that chloroform or ether extracts from acidulated aqueous liquids almost the whole of the above bitters. The subsequent treatment with ether-chloroform in alkaline solution is usually unnecessary, as the principles of calumba and colchicum are in part extracted by acid chloroform. Seeing that the bitter principles of hops are entirely precipitated by neutral acetate of lead, the presence of some hop-substitute is absolutely certain if the chloroform or ether residue has a marked bitter taste. This can be ascertained in the course of a few hours, by the simple method above indicated, and half a pint of the beer is amply sufficient for the purpose.

The presence of a hop-substitute being proved by the marked bitter taste of the chloroform or ether extract, it will of course be very desirable to ascertain its nature, and in some important cases this can, fortunately, be effected very satisfactorily. In others we may expect future investigation to afford the necessary assistance.

I have made special endeavours to ascertain the possibility of definitely recognising quassia, which is one of the most important of the hop-substitutes actually employed. For this purpose I prepared quassiin in a moderately pure state by exhausting quassia wood with hot water, and treating the decoction with acetate of lead and chloroform, in the manner recommended for beer. The quassiin was obtained with some difficulty in a distinctly crystalline state, and otherwise it presented a close general resemblance to the description of it given by other observers. The following characters and tests were specially verified.

Quassiin is intensely and persistently bitter, sparingly soluble in cold water, more readily in hot, and easily soluble in alcohol. Its best solvent is chloroform, which extracts it readily from acidulated solutions.

An aqueous solution of quassiin does not reduce Fehling's solution, or ammonio-nitrate of silver. The solid substance gives no coloration (or merely yellow) when treated with strong sulphuric acid, or with nitric acid of 1.25 sp. gr.; nor is any colour produced on warming. These four negative reactions are important; for *picROTOXIN* reduces Fehling's solution, and gives an orange-red colour with sulphuric acid; *gentiPICRIN* and *menyanthin* reduce ammonio-nitrate of silver, and the former gives a red colour, and the latter a yellowish-brown, changing to violet-red when warmed with sulphuric acid; and other bitters mostly give more or less characteristic reactions.

A solution of quassiin gives a white precipitate with tannin. The reaction is used by Christensen, Oliveri, and others to isolate quassiin from its solutions, and by Enders to separate it from *picROTOXIN*. In my hands the reaction has not proved satisfactory. The liquid is very difficult to filter, and the filtrate still retains an intensely bitter taste, showing that the precipitation is very incomplete. As an analytical method the reaction is useless, but it is of some value as a qualitative test.\* The test must be made in a cold solution.

Quassiin gives a brown coloration with ferric chloride. The reaction is best observed by moistening a quassiin residue in porcelain with a few drops of a weak alcoholic solution of ferric chloride, and applying a gentle heat. A fine mahogany-brown coloration is produced.

The most delicate and characteristic test for quassiin is based on an observation of Christensen. On treating quassiin with bromine a derivative is obtained, which is stated to be more bitter than the original substance. On adding caustic soda the bitter taste is said to be destroyed, but a product of a fine yellow colour is obtained. I am unable to confirm the destruction of the bitter taste, at least entirely, but the coloration is marked and characteristic.

The following is the best way of applying the test. The substance to be tested for quassiin is dissolved in a little chloroform, or if a liquid is agitated with chloroform,

\* Possibly more complete precipitation of quassiin by tannin could be effected in an alcoholic solution.



and the aqueous layer separated. The chloroformic solution is then treated with bromine water until the yellow colour remains after agitation, showing that the bromine has been used in slight excess. The aqueous liquid is then removed (or if small in volume may be neglected), and the chloroform agitated with ammonia. This produces immediate destruction of the colour due to the bromine, and if quassia be absent both the chloroform and ammoniacal liquid will be colourless. In presence of quassia the ammonia will be coloured a bright yellow.

The chloroform-residues from camomiles, calumba, colocynth, cocculus, and chiretta do not give any similar reactions with bromine and ammonia. The ether-residue from chiretta gives a straw-yellow coloration gradually changing to a dull purplish-brown, but the fact that no such reaction is yielded by the chloroform solution of the drug renders confusion with quassia impossible. Picric acid yields a solution in chloroform which is but slightly coloured compared with the deep yellow liquid produced on subsequent agitation with ammonia; but if its presence be suspected it can be readily and completely removed by agitating the chloroformic solution with soda or ammonia, and separating the alkaline liquid before employing bromine.

With a view of ascertaining how far the foregoing reactions of quassia were likely to be of service in practice, I added to one litre of a mild beer, which had been previously proved to yield no bitter principle to chloroform after treatment with acetate of lead, sufficient infusion of quassia to make a perceptible difference in the flavour. The liquid was concentrated, precipitated with neutral lead acetate, the filtrate treated with sulphuretted hydrogen, and the refiltered liquid further concentrated and agitated with chloroform. On evaporating the chloroform a residue was obtained which had an intensely bitter taste, and yielded a solution which gave a white precipitate with tannin, but did not reduce ammonio-nitrate of silver. The residue gave no colour on warming with concentrated sulphuric acid, but gave a well-developed mahogany-brown colour with ferric chloride. By the bromine and ammonia test it gave a strong yellow coloration.

The amount of residue obtained would have sufficed to obtain all these reactions several times, so that it may be considered established that *quassia can be detected with certainty and facility in a moderate quantity of beer containing it.*

The employment of *chiretta* as a hop-substitute has been repeatedly recorded by previous observers, but no tests are given for it by Dragendorff or others who have worked on the subject. I found it in quantity in two hop-substitutes I recently examined, and suspect its presence in a third. The active principle (chiratin,  $C_{25}H_{48}O_{15}$ ) is intensely bitter, sparingly soluble in cold water, rather more so in hot, and is readily dissolved by alcohol and ether, the latter solvent readily removing it from its aqueous solution. On the other hand, chloroform removes but little bitter principle from an aqueous infusion of *chiretta*. Chiratin is a neutral substance, decomposed by dilute acids into ophelic acid and chiratogenin. It does not reduce Fehling's solution, gives a copious precipitate with tannin, and is not precipitated by neutral lead acetate. The reaction of the ether-residue from infusion of *chiretta* with bromine and ammonia has already been described.

It is evident that our knowledge of the chemistry of the vegetable bitters available as hop-substitutes is very incomplete, and it is only by its further study we can hope to fully solve the problem of their detection in beer. But I believe we can already

distinguish with certainty and facility between "hops" and "not hops," and that ought to suffice in many cases. When we examine butter we are content to define the admixture as "foreign fat," and we make no attempt to specify the exact nature or origin of the foreign fat employed. I submit that we are fully able to take a similar position with respect to hops and hop-substitutes.

#### NOTE ON THE COMPOSITION OF SOME PREPARATIONS SOLD AS HOP-SUBSTITUTES.

By A. H. ALLEN AND W. CHATTAWAY.

*Read at the Meeting, May, 1887.*

THE detection of hop-substitutes in beer is so intimately connected with the question of the nature of the hop-substitutes actually employed, that we have thought it of importance to examine some of those preparations most extensively advertised. We have been struck with the great difference in the price and the physical appearance of the advertised hop-substitutes. Time has not allowed us to make so full an examination of the chief advertised preparations as we could have wished, but the following observations will be of interest.

In sample A, by examination with a lens, quassia, chiretta, and hops were recognised without difficulty. There was also present a seed not unlike that from a cruciferous plant, the exact nature of which was not identified.

Sample B was of a very extraordinary character. It consisted largely of rosin mixed with catechu or cutch, or some closely analogous tannin extract. Considerable quantities of fish-gelatin and chiretta were also present, and sodium sulphite was detected in addition. Whatever may be the value of fish-gelatin, sodium sulphite, and tannin, in the manufacture of beer, they can hardly be legitimately termed "hop-substitutes." The object of adding rosin is not very apparent; possibly it may have been an attempt to replace hop-resin, in which case truly a little knowledge is a dangerous thing. The proportions of the several constituents of this sample may be inferred from the following statement of the results of its treatment by solvents, etc.:—

Moisture, driven off at 100°C.	.. .. .	8.5 per cent.
Ether extract, consisting of nearly pure <i>colophony</i>	.. .. .	36.3 " "
Alcoholic extract, chiefly tannin and other constituents of <i>cutch</i>	16.6	" "
Aqueous extract	.. .. .	27.3 " "
Insoluble matter (by difference)	.. .. .	11.3 " "
		<hr/>
		100.0 " "
Ash	.. .. .	10.0 per cent.

We have examined the other samples of advertised hop-substitutes less completely. Quassia was distinctly recognised in the decoctions of two, and other bitter substances were also present. No picric acid or calumba was present in any case. Pyrethrum ("Persian insect powder") was almost certainly a constituent of one preparation.

A curious difference is observable in the prices charged by the manufacturers of



the advertised hop-substitutes. Thus the equivalent of 16 lbs. of hops was 3s., 4s., 4s., 4s. 2d., and 9d. respectively. So far as we can judge from the extent of our examination, the quality and suitability of the advertised hop-substitutes for their intended purpose is by no means to be gauged by the relative amounts charged for them.

With regard to the alleged injurious character of hop-substitutes we are not in a position to offer any personal opinion. Picrotoxin, the active principle of *cocculus indicus*, we all know to have marked poisonous properties, and we should probably agree in regarding picric acid as an objectionable addition to beer. It is within our knowledge that picric acid formed an ingredient of a well-known hop-substitute some years since. Of course, its detection in beer presents no difficulty. There is no recent authentic record of the use of picrotoxin in beer, but there are some curious facts which go in that direction. There is no legitimate use for *cocculus indicus* in the arts or medicine,\* and we are not aware of its receiving any application as a poison for the lower animals, except by fish poachers. According to Dragendorff, who was formerly chemist to the St. Petersburg police, *cocculus indicus* has been largely used for adulterating beer in Russia, and brewers have been frequently fined for the practice and the beer confiscated. Schubert, of Wurzburg, has stated that Bavarian beer has been often adulterated with *cocculus indicus*. We find the statement that in a discussion in the House of Commons (date not stated) Lord E. Cecil said that the quantity of *cocculus indicus* imported into England in 1857 amounted to 68 cwt.; in 1867, the quantity had increased to 689 cwt.; and "last year," that it amounted to 1064 cwt. These amounts are not large, and may possibly have received some legitimate application, but the greater part was probably re-shipped.

As to quassia, there seems to be a general opinion in its favour as one of the least objectionable of hop-substitutes. Systematic experiments with quassia are, however, almost wanting, except those of Campardon, referred to in the following extract from page 1264 of the "National Dispensary," by Stillé and Maisch:—

"Quassia is fatal to flies, and is sometimes used to preserve botanical collections from the ravages of insects. Rabbits and dogs have been killed by a concentrated preparation of the drug, the latter even by its application to the raw skin, and when its effects were not fatal it produced a partial paralysis of the limbs. Its bitter taste is more intense than that of most other stomachic tonics; like them, it excites the appetite for food and quickens digestion, but if too long continued it produces derangement of the stomach. Quassia may exhibit poisonous qualities, as in the following case:—A concentrated infusion of the drug was by mistake given in enema to a child four years old. Within an hour the child became unconscious and collapsed, the head was thrown back and the pupils were contracted, the respiration was inaudible, and the pulse could not be felt. It was restored by alcohol, ether, and ammonia (*Medical Record*, xviii. 404). The following conclusions were reached by Campardon in regard to quassia—crystallised and amorphous (*Bull. de Thér.* ciii. 385). In moderate doses it increases the secretion of the saliva, the bile, the urine, and perhaps of the milk. It stimulates the excretory ducts of the several organs producing these secretions. In appropriate cases of sickness it quickens the appetite, renews the strength, facilitates the excretions, renders defecation

\* It is an ingredient of an ointment which finds a very limited use.

easier, and hastens the expulsion of renal and biliary calculi. Its toxic action resembles that of the convulsing poisons.  $2\frac{1}{2}$  grains (gm. 0.15) of amorphous, or  $\frac{1}{4}$  grain (gm. 0.015) of crystallised quassia will occasion the following symptoms:—A burning in the throat and œsophagus, and a sense of constriction in the former; frontal headache; epigastric weight and pain; nausea, vertigo, confused vision, extreme restlessness and feverish impatience; unsteadiness of mind; frequent urination, loose stools, and vomiting; cramps and spasms in the muscles of the lower limbs."

Considering the small proportion of quassia which is required to impart a bitter taste to beer, it is questionable if it would ever be taken in practice in sufficient quantity to produce toxic effects.

We have ascertained by experiment that a solution of 1 part of crystallised quassia in 500,000 parts of water has a strong and persistent bitter taste.

#### DISCUSSION.

Dr. ADAMS said that he worked on the question of hop-substitutes some ten or twelve years ago, and he found no difficulty in distinguishing between the bitter of hop and the substitutes used for it. The method he found most useful was the precipitation by sub-acetate of lead, and there was no difficulty at all with the ordinary bitters such as quassia, calumba, gentian, chiretta, and wormwood, all remaining in solution, whilst the bitter of hop, and also the bitter of the camomile, which behaves like the hop, goes down and leaves the solution bitterless. He had specimens of all the hop-substitutes in use in England at that time, and without a single exception the solution remained bitter after treatment with the sub-acetate of lead.

After having separated the filtrate and evaporated it down, there was no difficulty in detecting the bitter; but he did not think there was any possibility of distinguishing between the individual bitters, considering the minute quantity present, and one could only positively say there was another bitter present besides that of the hop.

Dr. MUTER said that, with regard to the question of the detection of bitters in beer, he would say at once that he did not quite share the President's views as to there being no difficulty in detecting and identifying them. It was a subject that he had worked at in former years in connection with his book on *Materia Medica*. He had made numerous experiments himself, and he had repeated many of the published experiments, and he could prognosticate that, as the President went along, he would meet with several published reactions, which were partially incorrect and perfectly misleading. Although a few bitters would be found to be comparatively easy of detection—quassia, for instance, was a bitter which spoke very much for itself—many of them were, to a great extent, involved in difficulty and obscurity at the present time. There was no branch of chemistry that so much undesirable matter had been published about as that relating to *Materia Medica*, and many of the older researches were incorrect. No doubt one cause of this was that nowadays they had much better analytical appliances than the men who made these experiments. He did not for a moment mean to say that they were now cleverer than past observers, but for instance, they could not wash lead and other similar intractable precipitates in former days as one could now do by the aid of the filter pump, and the colour reactions for more than one proximate principle, which were given in books, were really not due to the principle at all, but to the traces of reagents and other matters that remained with them owing to the imperfect washing, which was almost certain to occur before the days of filter pumps. He was afraid that until the President, and others who might take the subject up,



had had time to work it out by the aid of modern appliances, it would be going too far to say they could swear positively that a sample contained no hop-substitute.

Mr. ALLEN said he thought they could tell whether it contained hops or a substitute for hops.

Dr. MUTER said then in that case how would they get on in cross-examination, seeing that they could not name the substitute? He did not remember how he first became acquainted with the lead process, but he believed that he could put his hands on it now. It must be quite thirteen or fourteen years ago since it was first published. Immediately it was brought out he had made experiments upon it, working on large quantities. Since that there had really been no other process that he knew of. He himself had used a process very similar to that mentioned, viz., precipitation with sub-acetate of lead, syphoning off the clear liquor after settlement, removing the excess of lead from this liquid, concentrating and tasting, and then extracting with immiscible solvents.

Now came a difficulty which shook his faith in his powers as an analyst as regards hop-substitutes. He had always believed in the process—from practising upon beer with various added bitters—until some time ago he got a beer which he was privately assured by the maker to have no bitter other than hops. This sample he put through the process, and he got a bitter out of that beer with chloroform after lead. He worked on a fairly large quantity, but the process here showed bitters other than hop, although he was assured that the sample represented as pure a beer as could possibly be obtained. Another difficulty was the quantity they might have to work on. Supposing an inspector brought them one-half or one-third of a pint, where was the process they could use? He had put bitters in the beer and worked on such quantities and failed to find them. In a case he knew of some time ago, there was some difficulty about some strychnine that was put in beer; he was aware of the very small quantity that had been put in because the chemist who had been stupid enough to lend himself to such a transaction had informed him of it. He made up some beer and divided it into two portions; tried for extraneous bitters in one portion by the regular beer way and there was not a sign of it; he then tried the other portion with a special toxicological process for strychnine and found it. He had, even then, to use eight ounces of the beer for this purpose, to get a really satisfactory ordinary reaction. In the present state of chemical knowledge, it would not be, in his opinion, safe to say they could detect any amount of added bitters to beer, however small, and go to the length of naming those bitters on the quantity they would have usually brought to them by an inspector. With a gallon of beer and an unlimited fee covering many days' work, they might, however, be able to do something satisfactory towards it.

Dr. DUPRE said he wished to protest strongly against the idea that a public analyst was always bound to make an analysis on only a few ounces of beer. He ought to have such a quantity as would enable him to certify the result with moral certainty. In his district he had only to write and get any quantity he wished for. No public analyst could be requested to make an analysis with a quantity which he himself believed to be utterly inadequate, and if any private person brings an insufficient quantity it is the analyst's plain duty to tell him so. As to strychnine, he might point out a case which occurred to him some years ago. A man, who had been wounded in India some time before, drank a glass of beer; he felt ill directly after, and went to a doctor, at whose house he had several tetanic convulsions. All he (Dr. Dupré) had to examine was the handkerchief with which the man had wiped his mouth, and the question was, had any strychnine been in the beer or not? He cut out some spots which looked like dried saliva, and worked upon them by the usual toxicological methods, but failed to detect

strychnine. He next added a minute fragment of bichromate to a portion of the suspected extract, and carefully evaporated it on a microscope slide, and finally, while looking at it with a 1-inch power, he added a drop of sulphuric acid, and the strychnine came out most beautifully. Referring to the microscope, Dr. Dupr  said that he was afraid chemists, as chemists, were not in the habit of using it as much as they ought.

DR. MUTER, in reference to Dr. Dupr 's remarks, begged permission of the chair to say that if the majority of public analysts acted in the way indicated by him, they would find themselves in very bad odour with the local authorities. If an inspector were told to always buy a gallon or two of beer, instead of the usual pint or quart, he would reply that the vendor would at once suspect him, and declare the article, and so the purchase would be rendered nugatory for the purposes of the Act, and the money of the public wasted. As regards the application of delicate and tedious toxicological processes to beer in every case, there would then have to be a revision of the fee of 10s. 6d. per analysis laid down by the Act. It was one thing to analyse directly for a specifically suspected poison, and quite another matter to generally examine an article for adulteration for 10s. 6d.

MR. SALAMON said that he should like to hear some views about the varying extractive influences of different brewing-waters upon hops. It was well known, for instance, that waters containing sulphate of soda had a different influence to waters containing sulphate of lime, and he thought it would be well for them, before dogmatising upon what would be a public question (because analysts would be called upon to distinguish these bitters), to ascertain, if possible, whether the varying salts in waters did exercise any influence upon the bitter itself. It might not be improbable that the case which Dr. Muter had mentioned fell within that category, and that it was an altogether different bitter which was extracted by the two waters.

With reference to the question of tannin, he might state, with accuracy, that not only had he to analyse a number of products coming from brewers, but he also had to go to the breweries and actually inspect the beer in process of manufacture.

In the year of the hop famine (1882) no doubt hop-substitutes were used to a large extent, but at present he did not know of a single brewery that used them, and it would be idle for them to do so with hops at the price at which they now were, but what they did use in a good many instances was tannin. He knew of some breweries where they used the most expensive hops they could buy; and yet, in producing a beer full flavoured, round on the palate, and low in alcohol, and in order to get the nitrogenous matter out of the beer, he had known extract of cutch, or catechu, to be used, for the purpose of precipitating the nitrogenous or albuminous matter, but that could not be termed a hop-substitute.

Another point they must bear in mind; he did not want to take a political side on the question at such a meeting, but the low price of hops was not produced by hop-substitutes, but by competition from abroad.

There was a general opinion among brewers, which was borne out by Dr. Graham, that hops had more to do with the drowsiness produced by beer drinking than alcohol, and there was consequently a tendency to reduce the amount of hops which they used, and to do that they sometimes used the cutch to which he had alluded.

In his opinion, this was rather a question as between brewers and hop-growers. If the brewers cared to come to a compromise with the growers—if the brewers like to say they will help their friends, the hop-farmers, then by all means let them do so.

They should be very careful in trying to compel brewers to use hops, whether they liked it or not. It would be absolutely impossible for them, as a body of chemical analysts, to prove that these hop-substitutes were more injurious than the hop itself.



Medical men would say that all these hop-bitters were less injurious to the system than the hop-bitter itself. It was evident from the present state of their knowledge that hop-substitutes are little used, and if used at all are harmless in beer, and he would recommend that the question should be threshed out as between the brewers and the hop-growers.

Mr. R. NORTON, M.P. for Tunbridge, as a visitor, expressed the great pleasure it had afforded him to be present, and, in reference to Mr. Salamon's last remarks, said that was not the position which was being taken up by the counties of Kent and Sussex. They felt that for a long time they were getting beer bittered with hops, but that now substitutes were used when hops were dear.

The generality of the public were under the impression that the beer was bittered with hops. It might be right or wrong, but at any rate they were under that impression, and in accordance with the Act the article should be supplied as demanded by the purchaser.

He wished that when a man went into a public-house, he should know whether the beer was bittered with hops or quassia; that was the point. He had listened with great pleasure to the debate there that night.

He, now, understood, first, that it was possible to say whether there was any other ingredient than the hop present, and secondly, if so, whether that ingredient was quassia or not.

They would now be in a position to take half a gallon of beer from any brewery, and say, "This is not bittered with hops." That was the main thing they had been contending for—they did not care whether analysts were able to say what the bitter was, whether camomile or gentian. They had reason to know that quassia has been very largely used.

Analysts could help him and his friends to that extent, if they were unable to come to the friendly compromise with the brewers which they were trying for. There were three or four millions of capital embarked in the hop industry, and they desired to know whether hops were wanted or not. If every time hops were over a certain low standard of value the brewers were to use substitutes, it was obvious that a crop which cost £40 an acre to produce, could not be continued under these conditions.

Mr. ALLEN, in reply: If he had an insufficient quantity of any sample, he certified that the quantity was insufficient for him to form an opinion.

He understood Mr. Norton to consider it of great importance that they should be able to distinguish between "hops" on the one side, and "not hops" on the other; this he believed could be done with certainty, ease, and on a very moderate quantity of beer. As to the identification of the various hop-substitutes, he did not profess to be able to distinguish all, but he thought he could already positively recognise calumba, quassia, colocynth, and some other bitters, including picric acid and picrotoxin, and if the matter became important he believed in a year or two public analysts would have devised methods for the detection of the other bitters, just as they had conquered other analytical difficulties when the occasion arose.

With butter they did not say with what fat it was mixed, but simply that it contained foreign fat; and they could do similarly with beer—that is, certify that it was beer; it contained a foreign bitter. As to tannin he would not admit that it was a bitter; it was an astringent, and if used in excess would simply make the beer nasty. It was not a hop-substitute; then why should it be used as an ingredient of a hop-substitute? It just showed that those gentlemen who were buying hop-substitutes did not get what they expected.

## REMARKS ON THE SO-CALLED ADULTERATION OF BEER, AND ON THE "BEER ADULTERATIONS," AND "BUTTER SUBSTITUTES" BILLS.

BY OTTO HEHNER.

*(Read at the Meeting, May, 1887.)*

IN consequence of the numerous prosecutions which have lately been instituted by the Inland Revenue Authorities, and which have resulted in convictions of London publicans and the infliction of heavy fines for selling watered beer, many public analysts have received communications from the Authorities appointing them, requesting explanations of their inactivity in a matter which is, both in Parliament and out of it, exciting a great deal of attention. Public analysts have hitherto been regarded as the custodians of the purity of all articles used for food or drink by man, and with them only rested the power to issue certificates of purity or of adulteration of legal value. The public, therefore, and the authorities appointing public analysts, are naturally surprised to see that in so important a matter as the control of the beer supply, the public analyst has been entirely supplanted by the Revenue officer, and the more so because the watering of beer is not a question which in any way touches the Inland Revenue. Under the Inland Revenue Act, 1880, the excise duty on beer is calculated as follows:—Upon every thirty-six gallons of worts of specific gravity of 1057, the duty of six shillings and three pence (less 6 per cent. for waste) is levied, and so on in proportion for any difference in quantity or gravity—that is to say, the brewer has to pay for each barrel of an original gravity of 1057 six and three pence, but for each barrel of 1028.5, only three and one penny half penny. He can brew strong or thin, as he pleases, without limit, provided he pays for each degree of gravity per barrel 1.316 pence. It cannot, therefore, be of any detriment to the Revenue if the publican reduces the strength of the beer by the addition of water, the duty on the gravity having been paid. The brewer commits no offence by adding water to his mash-tub in unlimited amount. How is it that the publican is held to offend grievously by doing that which is permitted to the brewer? It might be said that, in making, say, four barrels of brewery beer into five barrels of publichouse beer, the publican causes a loss to the Revenue to the extent of one barrel, but seeing that the brewer might have made, without contributing one single penny the more to the Revenue, and with perfect legality, five barrels out of the four, such an argument falls to the ground. As well might the thrifty housewife, who fills up the family teapot with hot water, after the first decoction has been withdrawn, be accused of defrauding the Revenue, so much less tea leaves, which pay a duty, having been used than in the ordinary course of tea-making.

Of course, the addition of sugar to beer, with or without the addition of water, is an offence against the Revenue. The sugar may ferment and form alcohol; it certainly represents an equivalent quantity of malt or other saccharine substance which should pay duty. But the addition of plain water is not a Revenue offence in any sense of the term. In the case of spirits, the duty is levied upon the percentage of proof spirit; no matter whether absolute alcohol or spirit of 35 U.P. is sold, the duty is the same for the same amount of absolute alcohol, and no Revenue offence is committed by the addition of water.



The addition of water to beer, therefore, can only be considered in the light of an adulteration, and as such should be punishable by the Sale of Food and Drugs Act. It is notorious that it is not so punishable, simply because beer, as permitted to be brewed, is a nondescript article, of no particular composition and of no particular strength. It is simply any liquor (Customs and Inland Revenue Act, 1885, sec. 4), "which is made or sold as a description of beer, or as a substitute for beer, and which on analysis of a sample thereof shall be found to contain more than 2 per cent. of proof spirit." That is to say, beer is anything that is sold as beer, provided it has 2 per cent. of proof spirit. If it is less strong, it ceases to be beer. Watered beer therefore is still beer, in the legal sense, and no offence is committed by watering, as the law at present stands. It would, no doubt, be an offence to sell, say, Bass's beer under that name after it had been diluted to one half its strength, because it would be no longer of the "nature, substance, and quality demanded;" but unless a sample is sold under a specific brand, adulteration of beer by water does not exist, as far as the powers of the public analyst, as defined by the Sale of Food and Drugs Act, are concerned.

It will be a matter of surprise to most public analysts that within the last two years an Adulteration Act was passed by Parliament, independent of the Sale of Food and Drugs Act, and without regard to the existence of public analysts—an Act which, on the face of it, has nothing whatever to do with the adulteration of food, but which nevertheless is a blow at the very existence of the public analyst. It is 48 and 49 Vict., cap. 51, "The Customs and Inland Revenue Act, 1885," 6th August, 1885. It deals with a variety of matters affecting the Customs, with allowances to British spirits exported or used in warehouse; definition of the term beer, as given above; an amendment in reference to brewers' entries; regulation of entries, by brewers, of sugar stores and accounts of sugar to be kept; and, lastly, *a prohibition against adulteration of beer by brewers for sale, and dealers and retailers of beer.* This clause states:—

(1) A brewer of beer for sale shall not adulterate beer, or add any matter or thing thereto (except finings for the purpose of clarification or other matter, or thing sanctioned by the Commissioners of Inland Revenue), before the same is delivered for consumption; and any beer found to be adulterated, or mixed with any other matter or thing (except as aforesaid), in the possession of a brewer of beer for sale shall be forfeited, and the brewer shall incur a fine of fifty pounds.

(2) A dealer in, or retailer of, beer shall not adulterate or dilute beer, or add any matter or thing thereto (except finings for the purpose of clarification), and any beer found adulterated or diluted, or mixed with any other matter or thing (except finings), in the possession of a dealer in, or retailer of, beer shall be forfeited, and he shall incur a fine of fifty pounds.

It will be seen that here, in this Inland Revenue Act, the term *adulteration* which was so carefully excluded from the Sale of Food and Drugs Act is boldly used.

As to the justice of such clauses, as they affect brewers and publicans, I do not allow myself an opinion; they are the law. But I have the very strongest feeling that by their passing, the officers which for twelve years have had to look after "every article used for food or drink by man"—the public analysts—have been unnecessarily slighted. The clauses being included in an Inland Revenue Act, the Inland Revenue

officers naturally carried them out, although it was not specifically stated who were to be the persons entrusted with the analysis of the samples in question. But the very use of the term *adulteration* should have led to the employment of the regular officers—the public analysts. Here was a grand opportunity for harmonious action between the Revenue officers and the public analysts. The Excise officers, with their right of entry, could have collected the samples as sold by the publican, and as stored in the cellar, just received from the brewery; they should have handed them over to the inspectors acting under the Sale of Food Act, for analysis by the public analyst of the district. But that opportunity, which we public analysts would have joyfully welcomed, was lost, and in the eyes of our employers and of the public a damage was inflicted upon us which no amount of explanation of ours is likely to entirely efface. I will not stop to enquire how it came that such a clause was passed without ever being submitted to public analysts; it certainly could not have been drawn up without consultation with the Somerset House Laboratory officers.

But it is not only the Revenue which requires protection, but very much more so the public. A satisfactory legal definition of beer and other similar beverages is a pressing necessity. As in the case of spirits, limits of minimum strength, or original gravity, should be laid down, and if such limits be over-stepped, be it by the brewer or by the publican, the beverage should be considered to be not of the substance, nature, and quality demanded. It would be easy, without injury to legitimate trade, to devise such limits. How far the materials used in brewing should be defined and limited is a question which I hope to see answered by to-night's discussion; my own feeling is that neither hop nor malt substitutes should be prohibited as far as they are known to be non-injurious, but that unless any sample sold be made with malt and hops as main ingredients, due notice should be given to the purchaser. Whatever the relative merits of malt and hops on the one hand, and saccharine substitutes or bitter herbs on the other, may be, the purchaser has a right to know what he is drinking, and to choose between what he often believes to be the only genuine article, and one made with so-called substitutes.

Until such a definition has been laid down it is idle to talk of beer adulteration. We should refuse, meanwhile, to be used as catspaws in the interests of hop-growers, maltsters, or veiled Protectionists.

The Customs and Inland Revenue Act, 1885, upon which I have commented, was the thin end of the wedge driven into the interests of public analysts. That wedge is getting considerably thicker in the several Bills relating to the manufacture of beer and sale of butterine now before Parliament, and unless we public analysts bestir ourselves we will find that we have lost the right to analyse samples of beer at all, and in many cases samples of butter. For the Bills on beer are silent as to the machinery to be set in motion for the detection of adulteration; informers are to be paid one-half of the fines recovered; not a word is said about public analysts; but, judging from analogy, I believe that it is intended to use Revenue appliances. In the whole of the discussion which is taking place, the public analyst is ignored, reference is made to Revenue officers who have nothing whatever to do with the question, but who nevertheless silently accept the office of advisers on the same without openly and honestly



declaring that the public analysts of the country are the proper authorities to be consulted on matters relating to analysis of butter and malt substitutes.

We fare not much better in the Butter Substitutes Bill, and the Oleomargarine Bill, in which Custom House and Inland Revenue officers are empowered to examine goods in transit, and to employ experts—of course *Inland Revenue experts*—in the case of urgency. Plainly the provisions of the Sale of Food and Drugs Amendment Act, 1879, relating to the examination of milk in transit, should be extended to the butter and butter substitutes, not the control be taken from public analysts and given to Somerset House.

If the Society of Public Analysts has any *raison d'être* at all, it has clearly the duty to prevent, by all means in its power, the contraction of analysts' rights and powers, and to take care that after twelve years of labour for the public benefit they be not overlooked when questions arise touching their very existence.

#### DISCUSSION.

Mr. ALLEN said that the author had made some strong remarks, but they all knew him, and that he felt what he said.

From the newspaper reports it appeared that a man could ask at a counter for half-and-half, and be legally served, but two beers must not be mixed beforehand. A man was recently fined £30 for mixing two beers. The reason why the Inland Revenue were so anxious to prove that a man must not mix two beers was simply that it was wholly beyond their power to tell whether it was beer and water, or a mixture of a weak with a strong beer.

All that of course would disappear if they fixed a minimum strength for beer the same as for spirits. The limits of strength for spirits laid down in the Sale of Food and Drugs Act Amendment Act of 1879 had been found to work exceedingly well, and a similar limit for beer, based on the original gravity, would protect the public against excess of watering, and the public would soon find out which public-houses sold stronger beer than others.

The addition of sugar to beer was purely a matter for the Inland Revenue authorities, and had no interest for public analysts. The promotion of the use of an excessive quantity of salt could be similarly prevented by fixing a maximum limit for the total chlorine, and this should not be too low.

All they wanted could be done by putting a clause in the Bill, defining beer. But it would be much better if analysts were asked to state their views before the committee after it had passed the second reading.

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The next meeting of the Society will be held at Burlington House on Wednesday, the 8th inst., when several interesting papers will be read.

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#### THE BUTTERINE BILLS.

The President of the Society (Mr. A. H. Allen) and Mr. Otto Hehner gave evidence before the Parliamentary Butterine Committee on the 20th ult.

## APPOINTMENT.

Mr. BERNARD DYER has been appointed Public Analyst for the City of Truro.

## BOOKS, &amp;c., RECEIVED.

ANNUAL Report of the Connecticut Experiment Station for 1886; American Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Chemistry for Beginners, by R. L. Taylor; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Druggist's Circular; Hospital Gazette; Illustrated Science Monthly; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Scientific American; Society of Arts Journal.

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# THE ANALYST.

JULY, 1887.

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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of this Society was held at Burlington House, on Wednesday, the 8th June, the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened the following gentlemen were declared to be elected: As member:—W. T. MacAdam, public analyst for Portobello. As associate:—T. W. Glass, assistant to Dr. T. Redwood.

The following papers were read and discussed:—

"Notes on the Logwood Test for Alum in Bread, etc.," by W. C. Young.

"Quantitative determination of Pepper Mixtures," by A. W. Stokes.

"Note on Pepper Adulteration," by T. Stevenson.

Dr. Muter's paper "On the American Methods of Manufacturing Oleomargarine, and the laws on the subject in the United States," was taken as read.

The President then referred to

## THE BILLS FOR DEALING WITH BUTTER SUBSTITUTES.

The PRESIDENT said:—

A Committee of the House of Commons is now sitting, under the presidency of the Right Hon. G. Selater-Booth, to consider the two Bills proposing to deal with butter substitutes that were published in the last number of THE ANALYST. As you are aware, the Council have had the Bills under very careful consideration, and Mr. Hehner and myself have attended the meeting of the Committee on the two occasions on which witnesses have been examined.

The first witness was SIR FREDERICK ABEL, who commenced by describing the process of manufacturing butterine, as he had seen it carried out in Holland. He drew

a sharp distinction between butterine and oleomargarine, stating that the former was the finished product and ordinary article of commerce, while the latter was merely a manufacturer's intermediate product, and not sold retail at all. In fact, he described butterine as a mixture of 50 to 60 per cent. of oleomargarine (the more fluid portion of animal fat), with ground-nut oil or sesamé oil, churned milk or strongly-flavoured butter, and annatto or other harmless colouring matter. By an oversight he omitted to mention salt. Sir Frederick considered the term "butterine" preferable to "oleomargarine" as a generic name for finished butter-substitutes. He considered butterine perfectly wholesome and unobjectionable. When asked some questions relating to the analytical examination of butter, the witness stated that he had had no experience in that direction, and recommended that the inquiry should be made of an expert analyst.

Mr. HERBERT P. THOMAS, the principal clerk of the Local Government Board in charge of the Public Health Department, was the next witness. He doubted the necessity for any special legislation on the subject of butter-substitutes, believing the existing laws were sufficient, provided that they were better administered. There was a great disinclination on the part of many of the local authorities to carry the Sale of Food Act into operation, and the fines inflicted were generally insufficient. He was not prepared to recommend that any minimum number of samples per 1,000 of population should be collected annually. The analysts had generally been exceedingly well-selected and performed their duties in a satisfactory manner. The witness thought the existing provisions respecting the declaration of the nature of mixtures were sufficient. He had known instances in which the last three letters of the word "butterine" had been covered up with a price label.

I was the next witness called. I handed in a draft bill, drawn up by me after consultation with the Council of this Society. This draft wholly omitted the clause relating to informers, made the labelling of all butter-substitutes sold retail compulsory, and rendered an invoice equivalent to a warranty. In place of the unwieldy provisions relating to the employment of Inland Revenue officers, the draft contained a clause exactly similar to that in the Sale of Food Amendment Act, dealing with examination of milk in transit.

Another clause of the draft bill was one which the Council agreed with me in considering of great importance, and which ran as follows:—

"In the event of any sample analysed under this Act being referred to the chemical officers of the Inland Revenue Department at Somerset House, under the provisions of the Sale of Food and Drugs Act, 1875, the chemical officers aforesaid shall, in their certificate, state whether the sample had, by keeping or other cause, undergone any change that would interfere with the analysis, or the correct interpretation of the results of the analysis, and shall set forth fully their analytical facts and figures, and the reasons for their conclusions; and, in the event of their being unable to confirm the certificate of the public analyst, shall also state definitely whether they are in a position positively to contradict his conclusion; and in the event of the certificate of the chemical officers of the Inland Revenue Department failing substantially to support the certificate previously given by the public analyst, the public analyst may insist on being examined, and the prosecutor may require the attendance, for cross-examination, of the officer or officers of the Inland Revenue Department who made the analysis, and the court shall take his or their evidence and that of the public analyst, into consideration in deciding on the case."

Unfortunately, I was allowed no opportunity of explaining the unsatisfactory nature



of the present system of reference to Somerset House, although it is evident that the successful working of any Act dealing with butter-substitutes will be largely dependent on this point. Sir Richard Paget, who held a copy of Dr. Bell's book in his hand, asked if it were not the case that the methods of detecting and determining butter-substitutes were based on the differences of specific gravity, of the percentage of soluble and insoluble fatty acids, and on the differences of the melting points. I replied that they were good as far as they went, but if relied on exclusively, 35 to 40 per cent. of adulteration might be overlooked. The method of Dr. Bell, based on specific gravity, which had done good service in its day, was only a rough test, and there were two other tests which, nowadays, no analyst would think of omitting. These were the methods of Koettstorfer and Reichert, the latter of which gave the sharpest existing distinction between butter and butter-substitutes. In answer to Sir Henry Roscoe I described these processes in outline.

Much of my examination was devoted to the question of the desirability of abolishing the word "butterine" in favour of "margarine" or "oleomargarine." I stated that all were equally unscientific, and that "butterine" being now a generally accepted name for butter-substitutes, I saw no reason for prohibiting its use, provided that a clause were inserted in the Bill making it illegal to cover up or hide in any way the terminal letters. I stated that, according to my recollection, "oleomargarine" was the original name given to the finished article or factitious butter, though it might now be employed to designate an intermediate product. Still, "oleomargarine" is recognised as the ordinary name for butter-substitutes in America, and has recently been made compulsory all over the United States. Hence we have the anomaly that an article will be exported from New York as "oleomargarine," and on arrival at Liverpool the same name, according to Sir Frederick Abel and Mr. Lovell, has ceased to be applicable to it, is not correctly descriptive of it, and the article henceforth becomes "butterine." Whatever name be ultimately legalised for finished butter-substitutes generally, it is evident that the name "oleomargarine" is not obsolete, and has not the limited application attributed to it.

In the course of my examination I expressed the opinion that special legislation with respect to butter-substitutes was desirable, as there was no other article of an exactly parallel nature, except factitious wine. Thus, mixed coffee, adulterated pepper, and watered milk, contained some of the article under the name of which they were sold; but butterine often contained no real butter, except perhaps the trifling proportion added to give it a characteristic flavour.

Our friend Mr. OTTO HEHNER was the next witness. He stated that the mixing of butter with butter-substitutes was a manufacturer's operation, and was rarely practised by the retailer, and in instances within his knowledge the retailer had been misled by the wholesale dealer as to the nature of the article supplied to him. Mixtures containing a considerable proportion of real butter had recently become very common, and these mixtures he would wholly prohibit, as much increasing the tendency to fraud. He would not allow real butter to be added to a butter-substitute, except in the trifling proportion in which it is employed to communicate a butter flavour. I do not know how the trade would regard this, but it would certainly get rid of all difficulties for the analysts. A similar provision is contained in the German Butter-Substitutes Bill now under consideration.

Dr. JAMES BELL, of the Inland Revenue Laboratory, was the first witness examined on the re-assembling of the Committee on June 7th. He considered the retailer should always be the person proceeded against, even if he could show that he had been deceived

by the wholesale dealer. He recommended that an inspector should have the power to go into any butter-store, either wholesale or retail, and take a sample for analysis of any article of the character of butter, which was not distinctly labelled by the name which might be eventually adopted for butter-substitutes. In other words, the failure to label a butter-substitute as such should be made an offence, even when no sale took place. This appears to me a very valuable suggestion, and it is satisfactory to find that it met the approval of a subsequent witness, Mr. Lovell, who spoke on behalf of the wholesale butterine merchants. Dr. Bell did not approve of the proposed employment of Inland Revenue officers, and saw no object in providing for the taking of samples in transit. He thought it unnecessary to insist on any general name for butter-substitutes, and would not take the responsibility of suggesting one.

Some questions were put to Dr. Bell by Mr. McLaren, relative to a letter of mine in the *St. James's Gazette*, describing the objections of public analysts to the present system of reference of perishable articles to Somerset House. The portion of my letter bearing more particularly on the point was as follows :—

“ But a far graver cause of the inefficiency of the Sale of Food and Drugs Act, especially with regard to milk and butter, is the position of the Somerset House chemists, who are appointed referees under the Act. These gentlemen are placed in a very disagreeable and invidious position. Thus, they are required to certify whether the condemnation of a sample by a public analyst was correct, when it very frequently happens that they are not in a position to certify either one way or the other. But, instead of stating this in their certificates, these gentlemen consider it their duty to give the benefit of any doubt which may exist in their minds to the vendor of the article. The wording of their certificates has commonly been that they were ‘unable to affirm that the sample in question was adulterated;’ thus leaving the magistrates to imagine that they affirmed it to be unadulterated, whereas what they actually meant was that, from decomposition or other circumstances, they could not form any positive opinion on the matter. That this is frequently the case is evident from the fact that it has often been five or six weeks before the sample in dispute has reached their hands. Naturally, under such circumstances, no reliable analysis of milk has been possible; and, although the referees have attempted to avoid the difficulty by making an allowance for change in the milk according to the number of days it has been kept, it is evident that, without a knowledge of the circumstances and temperature under which it has been kept, whether the bottle was wholly or partially filled, and whether the milk was originally watered or not, any such corrections are of a wholly worthless character. Similarly, Dr. Bell, the head of the Somerset House laboratory, has published figures which show that a genuine butter is liable, by keeping for six or seven weeks, to undergo such changes as to make the analysis unreliable. It is, therefore, much to be regretted that the chemists appointed as referees under the Act should not definitely state these facts in their certificates, instead of leaving it to be inferred that the analyst who analysed the milk or butter when in a fresh condition had been in error. I may point out also, with every personal respect for the Somerset House chemists, that the assumption that they are bound to give the benefit of any doubt to the defendants is not in accordance with their position as impartial referees. It is their duty to state fairly the result of their analysis, and whether from it they believe the milk to have been adulterated. If they cannot speak with certainty, they might speak of probabilities; and, if they cannot make up their minds at all, they should say so candidly, and leave the course of giving the benefit of any doubt to the



Bench in the usual manner, and not exercise the prerogative of mercy themselves.

"It must further be borne in mind that the number of cases in which the analysts' certificates are disputed, and which therefore are referred to the Somerset House chemists, are very small, amounting only to a dozen or two of all kinds per annum. Hence the experience of Somerset House of such kind of work is necessarily limited, and the chemists there have no incentive to keep themselves *en rapport* with the advance of food analysis, in which public analysts are so much interested. One of the practical effects of this is that the Somerset House chemists habitually express themselves 'unable to affirm the fact of the adulteration' of butter with butterine when it occurs to a less extent than 35 to 40 per cent. ; whereas public analysts are able to detect with absolute certainty something like half this proportion. Bearing in mind, however, the damage to his reputation caused by a failure of the Somerset House chemists to confirm his certificate, the ordinary public analyst is often compelled in self-defence to give a certificate on which no proceedings can be taken, when he is perfectly aware in his own mind that the sample should not be allowed to pass."

On the subject of this letter Dr. Bell gave the following evidence:—

"Q. 551. You have issued statistics yourself, I think, to show that genuine butter is liable by keeping six or seven weeks, to undergo such changes as to make analysis unreliable?—A. No, certainly not. If butter is kept for a considerable time it depreciates; but the extent to which it depreciates within the month that it requires to be sent up to us is so infinitesimal that it would not affect any analysis."

"Q. 552. Is it not the case sometimes that four or five weeks elapse before you make the analysis after the thing is first sent up by the public analyst?—A. No, not four or five weeks after we receive it."

"Q. 553. After it is taken, I mean?—A. I think that under the present law they are required to send it up within the month."

"Q. 554. Can butter be kept a month and not affected at all?—A. Not to any appreciable extent."

"Q. 555. Except as regards flavour?—A. Yes."

"Q. 556. For analysis it is just as good?—A. Practically the same."

This evidence makes one rub one's eyes to be sure of being awake! It is simply astounding that Dr. Bell should be so little conversant with that part of the Act for the working of which he is directly responsible as not to know that the interval of time between the purchase of a sample and his receipt of a portion of it in case of dispute is commonly six or seven weeks. I do not suggest for a moment that Dr. Bell desired to mislead the Committee, but his reply certainly had that effect. Dr. Bell seems to have confounded in an unaccountable manner, the provision in the Sale of Food Amendment Act, under which proceedings in the case of perishable articles must be commenced within twenty-eight days from the time of purchase, with the length of time before he receives a portion of a disputed sample. He wholly ignores the fact that after the issue of the summons a period of not less than seven days must elapse before the hearing, and that in disputed cases there is very frequently an adjournment of the hearing for a fortnight or longer before it is decided to refer the sample to Somerset House. And the twenty-eight days allowed for taking out the summons is by no means an excessive time in practice, seeing that many sanitary authorities meet only once a fortnight and do not allow proceedings to be taken except under a special resolution passed by them.

But if Dr. Bell was mistaken as to the provisions and working of the Act, what shall we say of his statement about the change of butter by keeping, in the face of the experiments he records on page 71 of his book on the "Analysis and Adulterations of Foods, Part II."? There he gives the following data, "showing the amount of depreciation which different samples of butter have undergone in the respective times stated":—

	1.	2.	3.	4.	5.	6.
Insoluble acids; original butter ..	87.30	87.80	85.50	87.40	87.72	87.65
" " after keeping ..	88.97	90.00	85.72	87.97	88.40	88.00
" " increase ..	1.67	2.20	0.22	0.55	0.68	0.35
Length of time kept, in weeks ..	12	7	7	6	8	6

In the most striking case, No. 2, a butter kept for seven weeks showed an increase of 2.20 per cent. in the insoluble acids, the specific gravity at 100° Fahr. at the same time falling from 911.58 to 909.19. It would be interesting to learn from Dr. Bell how in the face of such a result he can state that he has never issued statistics showing that genuine butter, by keeping for six or seven weeks, is liable to undergo such changes as to make analysis unreliable, and that the depreciation in a month is so infinitesimal that it would not affect any analysis. Of course all analysts know that such figures as were obtained by Dr. Bell, after keeping No. 2 sample for seven weeks, are such as indicate a notable adulteration, but having obtained them it is easy to understand how it comes that Dr. Bell fails to detect adulteration of butter even in cases where the admixture may exceed thirty per cent. Personally, I have no confidence in the figures in question, and doubt if butter is liable to undergo the rapid change found in the case of No. 2 sample; but Dr. Bell is in a different position. If he maintains the accuracy of his reply to Q. 551 it is a distinct repudiation of his analyses on page 71 of his book.

Dr. Bell's examination then proceeded as follows:—

"Q. 557. Is it the case that the wording of the certificate from Somerset House frequently is simply that you are unable to affirm that the sample in question is adulterated?—A. That is so in the case of milk; we have not a certificate in the case of butter worded in that form."

I confess that this reply surprised me, as I was not aware that the objectionable "inability to affirm the fact of adulteration" was limited to milk certificates.

"Q. 558. A certificate like that of course leaves the whole thing perfectly vague?—A. It leaves the magistrates to deal with the question."

"Q. 559. Does it not rather give a presumption against the local analyst?—A. No, I do not see that it does; the terms are rather in favour of the public analyst."

"Q. 560. I should have thought they were against him?—A. Decidedly not; it leaves the magistrates either to accept our certificate or not."

"Q. 561. I am informed that the local analysts feel that that phrase is rather against them, and that, in consequence, public analysts are often compelled to give certificates on which no proceedings can be taken for fear of your sending down this vague report, when, in reality, they are perfectly aware in their own minds that the sample should not be allowed to pass?—A. The public analysts are pretty well aware



now-a-days that they need have no dread of our interfering with their analyses, provided they have kept within a reasonable limit and made an accurate analysis."

"Q. 562. Then you think that no damage is done to the public interest by long delay in getting the Somerset House decision.—A. No."

I intend in my next batch of quarterly reports to state that Dr. Bell considers the expression "we are unable to affirm the fact of adulteration" of milk to be rather in favour of the public analyst, and that it leaves the magistrates either to accept the Somerset House certificate or not. I should recommend all other public analysts to follow my example.

Mr. JOHN CARY LOVELL was the next witness. He said he had had great experience as an importer and distributor of butter-substitutes. He restricted the term oleomargarine to the manufacturer's intermediate product. He thought the sale of butterine for butter should be put a stop to. He apparently had every confidence in public analysts, and approved of Dr. Bell's suggestion that an inspector should be allowed to take, without purchase, samples of apparent butter not having a butterine label. He objected to the suppression of the word butterine. All butter-substitutes sold retail should be wrapped in descriptive labels, and the word "butterine" should be branded on all kegs and cases. By inflicting sufficiently heavy fines, and even imprisonment for repeated offences, the fraudulent sale of butter-substitutes would soon cease. He would apply the term butterine to everything but genuine butter.

The Committee will meet again on June 10th, when representatives of the wholesale and retail butter-trade will be heard.\*

Speaking generally, the provisions of the Bills which public analysts thought most objectionable have little chance of becoming law, and if the suggestion of Dr. Bell with respect to taking samples without purchase be adopted, the Act will be of decided public benefit.

The attention of the Committee has been directed by letter to the method of dealing with suspected milk in Derbyshire, for which county Mr. Hehner and myself are joint analysts. In the case of either of us having a sample of milk believed to be adulterated, he sends it without delay to the other, who also gives a certificate on it (at half fee), so that there are two independent analyses made of the fresh, or nearly fresh, milk. This course practically precludes all chance of error on the part of the analyst, and is a far more satisfactory way of checking him than by sending the milk to Somerset House, when five or six weeks old, and when, therefore, it is quite impossible to make any trustworthy analysis of the sample. As an illustration of this I may state that in a recent case an analyst certified to 16 per cent. of added water in milk, and his certificate being doubted, the sample was sent to Somerset House, whence a certificate was returned stating that the milk was adulterated with 40 per cent. of added water! The discrepancy is an indication of the value of the Somerset House "allowance for change by keeping."

Mr. HARLAND said that there are in London makers of oleomargarine simply, and other firms who mix oleomargarine with oil or milk and produce the article butterine. Recently he had had to examine a number of butters as they came into London. A good many brands had been warranted genuine and suspicion had arisen about them.

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\* The witnesses examined on June 10th gave evidence merely of trade interest.

As a matter of fact, more than 50 per cent. of them contained from 30 to 60 per cent. of butterine; they were branded or warranted genuine and had the name of a respectable trader attached.

Dr. MUTER said he could confirm the President's recollection that the term oleo-margarine was originally applied to the finished product, and not limited to the intermediate article as it now is by some traders.

The President announced that the country meeting of the Society would be held at Leamington, on Friday, the 22nd inst., and full particulars would be duly announced.

The papers read as above noticed will be published in *THE ANALYST* for next month.

#### VISIT OF THE SOCIETY TO THE FARMS OF THE AYLESBURY DAIRY CO.

On Thursday, the 9th June, the members of the Society who were in London, for the previous evening's meeting, journeyed into Surrey, to visit the above Company's dairy farms, when a very interesting day was spent. A full account of this visit will appear in our columns during the recess.

#### TESTING SKIM-MILK BY THE LACTOCRITE.

By HARALD FABER.

*Read at the Meeting, April, 1887.*

In a previous paper on the Lactocrite I tried to prove how very exact results may be obtained when determining the amount of fat in whole milk by means of this apparatus, and how easily it is worked. I mentioned that the results obtained, when testing skim-milk, would fall too low, and by two examples I showed that the difference is about 0.1 per cent., when the skim-milk contains about 1.0 per cent. of fat. I shall now give the results of some experiments on milk containing still less fat, viz., skim-milk from very close hand skimming and from cream separators. I mentioned that the differences between the Lactocrite and analysis are found to be about 0.2 per cent. for separated milk, the result of a series of experiments carried out in Sweden being:—

Chem. Analysis.	Lactocrite	Difference.
0.50	0.30	0.20
0.50	0.29	0.21
0.43	0.20	0.23
0.36	0.10	0.26
0.34	0.12	0.22
0.26	0.05	0.21
0.19	0.00	0.19
0.19	0.00	0.19

My own experience showed me that sometimes the difference was still larger, sometimes also less, making the use of the Lactocrite for testing separated milk rather doubtful. Besides, when the Lactocrite shows no fat, it is impossible to know how small an amount has been left by the skimming. In one case I found, in testing a milk containing 0.31 per cent. of fat, that the Lactocrite showed only a trace which could not be



measured, so that separated milk with less than 0.3 per cent. of fat can not be tested at all.

The reason why the very small amounts of fat, so to speak, disappear, I cannot give. It might be imagined that the boiling with acid would attack the small globules, and in separated milk only the very smallest globules are left; but if that was the case it would seem very strange that so exact results are obtained with whole milk, as the size of the globules varies much in different samples of milk, and especially in milk from different breeds. The following experiment seems also to controvert this theory:—

Six samples of the same separated milk, containing 0.49 per cent. of fat, were tested in the Lactocrite, three of them being boiled with acid for only four minutes, the other three for eight minutes. The results obtained were as follows:—

	Boiled 4 min	8 min.
Per cent. of fat found	0.2	0.2
"	0.2	0.25
"	0.25	0.25

If the acid did attack the fat, less fat ought to be left after the longer boiling with acid, which was not the case.

It may seem of little interest to be able to estimate such small amounts of fat with very great accuracy, but in butter dairies it will be found of the greatest importance. Farmers look for help and information from agricultural chemists, and chemical analysis can hardly benefit the dairy farmers more than by teaching them how to skim the milk thoroughly with a separator, if they go in for buttermaking; as by a well-worked good separator the milk may be made to produce 20 per cent. more butter than by the old system, in some cases even considerably more. But this is only done when the separator is made to skim the milk thoroughly, which alone can be controlled by a test of the amount of fat left in the skim-milk.

Seeing how great a service the Lactocrite might yield the buttermaker, if it could be made to test the separated milk with sufficient exactness and in a simple way, I tried to alter the method of working, and finally found that by using it in the same way as the "Control Centrifuge" of Professor Fjord (see ANALYST of January) very satisfactory results could be obtained.

The method I use is still easier than the ordinary working of the Lactocrite. All that is required is to fill the test-boxes with the skim-milk until it reaches about half-way up on the graduated glass, and then place them in the warm disc and make this revolve. It takes a longer time for these very small globules to rise in milk not treated with acid, so that separating for twenty or thirty minutes will be necessary. After that time a column of cream is found in the graduated glass, which it is very easy to read off. This cream, however, does not directly give any information of the amount of butterfat in the skim-milk; a calculation is necessary. I have found that multiplying the degrees of cream [each degree corresponding to 0.1 per cent. of fat when testing whole milk] by the factor 0.03 gives the per centage of fat in the skim-milk with remarkable correctness. Below are the results of fourteen samples, resulting from eleven different separatings of different milks, separated by different separators:—

Per cent. of fat by Analysis.	Degree of cream by Lactocrite.	Degree of cream × 0.03.	Difference.
0.08	2½	0.075	0.005
1.01	1	0.03	0.08
0.13	4	0.12	0.01
0.22	7	0.21	0.01
0.24	7½	0.225	0.015
0.25	8	0.24	0.01
0.28	9	0.27	0.01
0.31	9½	0.285	0.025
0.31	11	0.33	0.02
0.32	13	0.39	0.07
0.35	15	0.45	0.10
0.54	16½	0.495	0.045
0.55	18½	0.555	0.005
0.76	22½	0.675	0.085

Average 0.007

Numeric average 0.035

Only in four cases is the difference larger than 0.05, which I consider the difference to be guaranteed by chemical analysis, if this shall claim to be exact.

It is, perhaps, not so very strange that tolerably uniform results should in this way be obtained by a test, based on the "cream," which is otherwise not a very uniform product. For, in the first place, the centrifugal force, when, acting strongly for half an hour, is well able to cause all the globules to rise, and pack them closely, and secondly, these globules, including (in separated milk) only the very smallest, are of much more uniform size than the globules in whole milk. At any rate the number of requirements seems sufficient to prove that, worked in this way, the Lactocrite may be used as a practically reliable test for skim-milk. By means of the Lactocrite, therefore, a dairy farmer may examine the working of a separator before buying it, and he may at any time test whether his milk is skimmed so closely as to give him the largest possible yield of butter, since he will be able to perform in a short time a sufficiently correct analysis of his skim-milk at very little cost. By this machine, therefore, may be established a control which hitherto has been largely neglected.

*Conclusion of the Society's Proceedings.*

#### A METHOD FOR THE SEPARATION AND ESTIMATION OF BORIC ACID.

*With an Account of a Convenient Form of Apparatus for Quantitative Distillations.*

By F. A. Gooch.\*

(Continued from page 94.)

To bring the matter to the test, the following experiments were made. In them and in all succeeding experiments the boric acid was weighed in solution, the standard of this having been fixed by dissolving in a known weight of water a known weight of fused boric anhydride prepared in a state of purity by frequent recrystallisation. The magnesium oxide employed was made from the pure chloride by precipitating by

\* American Chemical Journal.



ammonium carbonate and igniting, and was free from lime and alkalies, and, as far as could be determined, otherwise pure. The whole operation of each experiment was conducted in one vessel, so as to avoid transfers. In all cases a weighed platinum crucible of 100 c.c. capacity received a weighed portion of magnesia, and after ignition and subsequent weighing the weighed solution of boric acid was introduced. In experiments (1) to (4) the magnesia was thoroughly stirred in the solution of boric acid, the evaporation carried at once to dryness, and the crucible and residue ignited and weighed; in experiments (5) to (8) the magnesia was dissolved, after the addition of the boric acid, in hydrochloric acid sufficient in amount to prevent the precipitation of magnesium hydrate on the subsequent addition of ammonia, ammonia introduced in considerable excess in (7) and (8), in distinct excess in (5) and (6), the whole evaporated and ignited, the residue moistened and again ignited, and this last treatment repeated until the residue ceased to yield vapour of hydrochloric acid when heated.

	B <sub>2</sub> O <sub>3</sub> taken. Gram.	MgO taken. Gram.	MgO + B <sub>2</sub> O <sub>3</sub> found. Gram.	B <sub>2</sub> O found. Gram.	Error. Gram.
(1)	0.1734	0.5005	0.6607	0.1602	0.0132—
(2)	0.1804	0.4973	0.6660	0.1687	0.0117—
(3)	0.1793	0.4949	0.6640	0.1691	0.0102—
(4)	0.1794	0.4941	0.6627	0.1686	0.0108—
(5)	0.1807	0.4984	0.6542	0.1558	0.0249—
(6)	0.1789	0.4974	0.6687	0.1560	0.0229—
(7)	0.1806	0.4944	0.6684	0.1740	0.0066—
(8)	0.1789	0.4959	0.6672	0.1713	0.0076—

From these results it appears plain that under the conditions of the experiments neither magnesia alone nor the magnesia mixture is efficient in fixing boric acid; but in experiments (7) and (8), in which ammonia was employed in large excess, the loss of boric acid is least, so that it would seem to be the case that though ammonia is not a perfect preventive of volatilisation, it does exert a restraining action on the boric acid. That the magnesia mixture should be incapable of retaining entirely the boric acid present is, as has been pointed out, not surprising; but that the loss should be so great is rather startling, and more than suggests that the errors of Marignac's process are seriously excessive. The failure of magnesium oxide to hold back boric acid under the conditions of the experiment must be due to a cause other than that which determines the loss during the evaporation and ignition of the magnesia mixture, and for this it is natural to turn to the insolubility of the oxide—a quality likely to oppose some difficulty in the way of establishing complete contact between the boric acid and the magnesia during a short exposure. Direct tests of this point showed distinctly that mixtures of boric acid in water and magnesia, when submitted at once to distillation, yielded boric acid to the distillate; but that, if the mixture were permitted to stand some hours before distilling, the oxide passed to the semi-gelatinous condition of the hydrate, and retained the boric acid so firmly that turmeric failed to show the presence of the latter in the distillate. It is plain, therefore, that with sufficient preliminary exposure magnesia might be relied upon to retain boric acid; but inasmuch as long and perhaps somewhat indefinite periods of waiting are objectionable in any analytical process, it was thought best to try the effect of substituting lime for magnesia. Experiments (9) to (12), con-

ducted like the previous ones, excepting only the use of carefully prepared and ignited calcium oxide instead of magnesium oxide, were made with this end in view.

	B <sub>2</sub> O <sub>3</sub> taken. Gram.	CaO taken. Gram.	CaO + B <sub>2</sub> O <sub>3</sub> found. Gram.	B <sub>2</sub> O found. Gram.	Error. Gram.
(9)	0.1810	0.9737	1.1560	0.1823	0.0013 +
(10)	0.1819	0.9750	1.1583	0.1833	0.0014 +
(11)	0.1808	0.9922	1.1810	0.1818	0.0010 +
(12)	0.1833	0.9715	1.1560	0.1845	0.0012 +

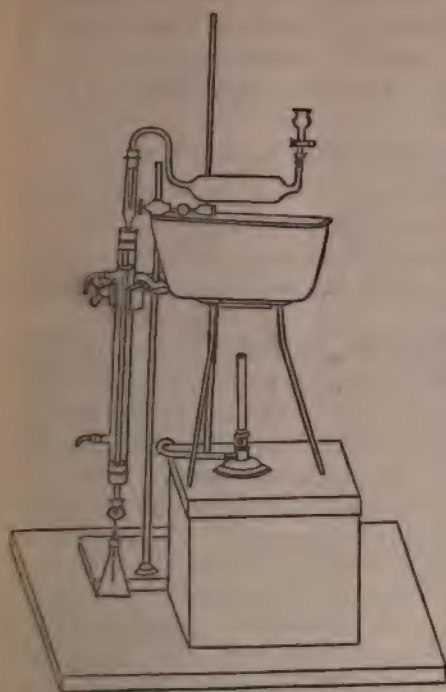
These figures indicate sufficiently that there is no loss of boric acid by volatilisation when its aqueous solution is evaporated in contact with calcium hydrate; but, inasmuch as the comparative solubility of the latter is the quality which makes it effective where magnesia is not, it seemed desirable to test the action of calcium hydrate in alcoholic solutions, in which it is very insoluble. The experiment showed that when the solution of boric acid in methyl or ethyl alcohol is put upon lime and distilled at once, loss is apt to take place, and sometimes to a very considerable amount, but that a short period of digestion, with occasional stirring—from five to fifteen minutes—is sufficient to obviate danger of volatilisation of boric acid.

It appears, therefore, that, free boric acid being easily volatilised by means of methylalcohol and fixed completely by calcic hydrate, the separation of the acid from almost everything with which it occurs ordinarily, and its estimation subsequently, depend only upon the practicability of distilling it from its compounds in such company that it may be retained by lime, and its amount determined by the increase in the weight of the latter. Unlike magnesium chloride, calcium chloride does not yield its chlorine readily under the action of heat and moisture naturally retained; so that hydrochloric acid must not be present with boric acid, which is to be estimated in the manner described. Calcium nitrate and calcium acetate both yield the oxide without difficulty upon ignition, and nitric and acetic acids are suitable agents, therefore, for the liberation of boric acid previous to distillation.

The actual distillation presented at first some difficulty—for the repeated, thorough, and rapid, evaporation of a liquid charged with soluble or insoluble solid matter is apt to involve some mechanical transfer to the distillate of material which should remain in the residue—but the device of the following description solves the problem successfully.

The apparatus, which is shown in the accompanying cut, consists essentially of a retort, condenser, and bath for heating. For the last I have used a paraffine bath, as being on the whole the most convenient. The condenser is set vertically, to facilitate changing the level of the retort within the bath, and to secure at the same time continual and thorough washing of the tube by its own condensations. The retort, somewhat like the well-known drying-tube of Liebig in general shape, is easily made of a pipette by bending the tube at one end to a right angle, at the other to a goose-neck, as shown. To the former end is fitted, by a rubber stopper or section of tubing, a glass funnel-tube provided with a stopcock; the end of the goose-neck passes tightly through a rubber stopper in the upper end of the condensing tube. This is essentially the apparatus, but it is convenient to attach to receive the distillate a small Erlenmeyer





flask, which moves with the condenser and is joined to it, in the manner indicated in the figure, by means of a thistle-tube and a rubber stopper, grooved to permit the free passage of air. In carrying out a distillation, the liquid to be distilled is introduced into the retort, either by the funnel-tube or previous to its insertion; the glass cock is closed, the water started through the condenser, and the retort lowered into the hot paraffine, care being taken to begin the operation with the retort not more than half full, and so inclined that only the rear dips below the surface of the bath. If the precaution to heat the retort at the start in this manner be overlooked, it may sometimes happen that the sudden and violent expulsion of air through the liquid will carry portions of it bodily into the goose-neck, and even into the condenser. With this point considered, the remainder of the operation presents no difficulty, and requires little care.

The size of the retort may be suited, of course, to the particular case in hand, but for most purposes a 200 c.c. pipette makes a retort of convenient dimensions, neither too large for the distillation of small charges nor too small to permit the treatment of 100 c.c. of liquid comfortably. The tube of the goose-neck should be wide enough to prevent the formation of bubbles in it; 0.7 c.c. is a good measure for the interior diameter. It is of advantage to heat the bath to a point considerably above the temperature at which the liquid which is to be distilled boils—something between  $130^{\circ}\text{C}$ . and  $140^{\circ}\text{C}$ . does very well for water, and is not too high for methylalcohol—and under such circumstances, and when the retort is entirely submerged, it often happens that evaporation takes place with extreme rapidity from the surface of the liquid in perfect quiet without actual boiling.

With such an apparatus the following experiments were made:—

The boric acid was weighed as before in solution, and, to bring the condition of the experiment to that of an actual analysis, 1 grm. of pure sodium hydrate was added in solution, nitric acid or acetic acid to acidity and a little more, and the whole was introduced into the retort and distilled to dryness.

In those experiments in which nitric acid was employed, the methylalcohol was introduced upon the residue thus dried in six successive portions of 10 c.c. each, and distilled to dryness; but in order to break up the residue of sodium nitrate, which by its insolubility might effect to some extent the protection of the boric acid from the action of the alcohol, 2 c.c. of water were introduced and evaporated between the second and third, and again between the fourth and fifth, distillations.

When acetic acid was made use of to free the boric acid, the six distillations with

methylalcohol were made as before; but sodium acetate being soluble in methylalcohol, the intermediate treatments with water were unnecessary. With the fourth portion of methylalcohol a few drops of acetic acid were added to preserve the acidity of the residue, which, as has been pointed out, tends to become alkaline under the treatment.

The residues of both processes of treatment were found to be free from boric acid, by the exceedingly delicate test with turmeric, care being taken, in the series of experiments in which nitric acid was used, to oxidise nitrates by means of bromine (expelling the latter before making the test), and in the acetic acid series to acidify with hydrochloric acid sufficiently to counteract the tendency of the acetate by itself to brown the turmeric on evaporation.

The lime to retain the boric acid in the distillate was ignited in the crucible in which the evaporation of the distillate was to be made subsequently, and then transferred to the receiving flask attached to the condenser, so that the boric acid might be fixed during the distillation. To prevent the caking of the lime by the action of the alcohol, it was slaked with a little water before the distillation was begun.

In experiments (13) to (16) nitric acid was employed, and in (17) to (20) acetic acid was used, with the precaution noted, to liberate the boric acid.

	B <sub>2</sub> O <sub>3</sub> taken. Gram.	CaO taken. Gram.	B <sub>2</sub> O <sub>3</sub> + CaO found. Gram.	B <sub>2</sub> O <sub>3</sub> found. Gram.	Error. Gram.
(13)	0.1738	0.9647	1.1392	0.1745	0.0007 +
(14)	0.1806	0.9639	1.1456	0.1817	0.0011 +
(15)	0.1779	0.9665	1.1450	0.1785	0.0006 +
(16)	0.1824	0.9739	1.1587	0.1848	0.0024 +
(17)	0.1806	1.4559	1.6371	0.1812	0.0006 +
(18)	0.1812	0.9720	1.1543	0.1823	0.0011 +
(19)	0.1788	0.9986	1.1781	0.1795	0.0007 +
(20)	0.1813	0.9527	1.1358	0.1831	0.0018 +

In experiments (13) to (16) the mean error amounts to 0.0012 + gm.; in experiments (17) to (20) the mean error is a little more than 0.0010 + gm. Throughout the entire series of experiments the tendency to yield figures slightly larger than the truth is manifest, but the error is quite within legitimate limits. The greatest care was taken to secure similarity of conditions under which the crucible and lime were weighed, before and after the evaporation and absorption of boric acid, and the weight after ignition was taken in every case after cooling over sulphuric acid during a definite period of ten minutes, in order to eliminate as far as possible the effect of atmospheric condensation upon the large surface of platinum. Ignitions were always finished over the blast-lamp, and constancy of weights secured.

The results of both modes of treatment are on the whole satisfactory, and equally so.

In the presence of chlorides, it is of course impossible to employ nitric acid to free the boric acid. Oxalic, citric, and tartaric acids also liberate hydrochloric acid to a considerable extent from alkaline chlorides. It was found, however, that when acetic acid was distilled over sodium and potassium chlorides, only traces of hydrochloric acid passed



into the distillate, and experiments (21) to (23) were made to determine whether these amounts are sufficient to vitiate the separation of boric acid from alkaline chlorides by distillation in presence of free acetic acid. The details of treatment were identical with those of experiments (17) to (20), excepting only the addition of 0.5 grm. of sodium chloride to each portion before distillation.

	B <sub>2</sub> O <sub>3</sub> taken. Gram.	CaO taken. Gram.	B <sub>2</sub> O <sub>3</sub> + CaO found. Gram.	B <sub>2</sub> O <sub>3</sub> found. Gram.	Error. Gram.
(21)	0.1834	0.9842	1.1675	0.1833	0.0001—
(22)	0.1831	0.9755	1.1593	0.1838	0.0007+
(23)	0.1761	0.9740	1.1523	0.1783	0.0022+

The mean error of these results is about 0.0009+ grm., and it is plain that the presence of sodium chloride does not materially change the conditions of the experiment. There seems, therefore, to be no reason why boric acid may not be separated by distillation from alkaline chlorides in presence of free acetic acid; but it was found that the presence of any considerable amount of potassium acetate is disadvantageous. Sodium acetate to a reasonable amount does not interfere with the favourable progress of the separation; but potassium acetate appears to require a much higher temperature for the expulsion of its water, and longer distillation.

When, therefore, chlorides are present in the salts from which boric acid is to be removed by distillation, the choice is open between two methods: The distillation may be made directly with an excess of acetic acid; or the hydrochloric acid may be first removed by means of silver nitrate, and the distillation of the filtrate proceeded with, at once, or after precipitation of the excess of silver salt by means of sodium hydrate or carbonate, care being taken to acidify again sufficiently with nitric acid after the removal of the silver. Of these two modes of proceeding, I incline to the treatment with nitric acid, and the removal of the chlorine by precipitation; and this method has been used with success by others as well as myself, for some months, in the analysis of waters carrying boric acid, and natural borates.

The process in either modification is fairly accurate and easily executed, and admits of very wide application. Insoluble compounds in which the boric acid is to be determined may be dissolved in nitric acid at once, or, if necessary, first fused with sodium carbonate; and, fortunately, nearly everything which is volatile in the subsequent treatment, and capable of forming with lime compounds not easily decomposable by heat, may be removed by known processes. The combination of fluorine, silica, and boric acid is perhaps most difficult to treat; but the precipitation and removal of the first as calcium fluoride from the aqueous solution of a fusion in alkaline carbonate may, it is believed, be effected with care, and the mode of procedure from that point is simple.

The number of distillations necessary depends, of course, upon the amount of boric acid treated. To remove 0.2 grm. of boric anhydride completely to the distillate, six charges of methylalcohol, of 10 c.c. each, proved, as we have seen, to be ample.

The apparatus, by the aid of which the distillation processes which have been described were carried out, has found useful application in a number of other processes. In the determination of free and albuminoid ammonia in waters which can be boiled

quietly with difficulty, in the methods of estimating hydrofluoric acid which involve the expulsion of silicon fluoride from a mixture of the fluoride with sulphuric acid and silica, in the separation of iodine from bromides and chlorides by distilling with ferric sulphate and sulphuric acid, and of bromine from chlorides by means of permanganic acid, it has proved of value, and will doubtless be found convenient in many analytical processes in which quantitative separations by the distillation of liquids liable to spatter or boil explosively are involved.

#### A NEW PROCESS FOR THE QUANTITATIVE ESTIMATION OF STARCH.\*

By PROF. DR. ALEXANDER V. ASBÓTH.

I HAVE often had occasion to try the various processes for estimating starch, and at one time used the process of Allin, until Märcker and Delbrück pointed out the results obtained by direct inversion are too high, as cellulose is also partly converted into sugar. Märcker proposed in 1885 to first heat the cereals with diastase, which, under favourable conditions, rapidly liquefies the starch. The filtered fluid may then be inverted by hydrochloric acid. In theory this process seems very convenient, but in practice it is very difficult of execution. First of all, the cereals must be finely ground, which with maize is a very difficult matter; then the liquid must be rapidly filtered whilst still hot, otherwise particles of starch will again separate out on cooling, so the operator is always in doubt whether all starch is in the filtrate. A single experiment is, therefore, not to be depended on, but from three to four trials must be made, which takes too much time. The great error in all these methods is, however, caused by the necessity of inverting the starch, which is always attended with loss. Franke has already, in 1882, called attention to the partial decomposition of starch when heated under a pressure of 3—3·5 atmospheres. Maltose gave a loss of 7·4 per cent. My own experiments also prove the decomposition of sugar, both by high pressure and by inversion. For these experiments I have used pure glucose (99·84 per cent.). On treating this first, according to Märcker's process, and boiling for two hours and a half, there was a loss of 3·84 per cent. sugar.

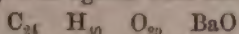
In a second experiment, I first boiled with 30 c.c. of a 1 per cent. solution of lactic acid, under a pressure of 3—3·5 atmospheres, then again with 200 c.c. of water, and 20 c.c. of hydrochloric acid, when the loss amounted to 8·54 per cent. Messrs Conrad and Guthreit have proved that dextrose, and more still, levulose, are completely decomposed by prolonged boiling with 7 per cent. hydrochloric or sulphuric acid in humic acid, acetopropionic acid and formic acid. After warming 9·5 grammes of cane-sugar with 800 c.c. water and ·72 grammes hydrochloric acid for half an hour, Soxhlet did not notice any loss, but on boiling for some time he found a serious diminution of sugar. *It is therefore plain no satisfactory determination of starch can be made by any process based on inversion.*

I therefore tried my best to estimate the starch by a direct process, and took advantage of a reaction first noticed by Zulkowsky, viz., the behaviour of starch towards baryta water, which yields an insoluble precipitate. My first efforts were now directed to ascertain the actual composition of this body, also to see whether it is constant. I

\* Repert. Anal. Chemie, No. 20.



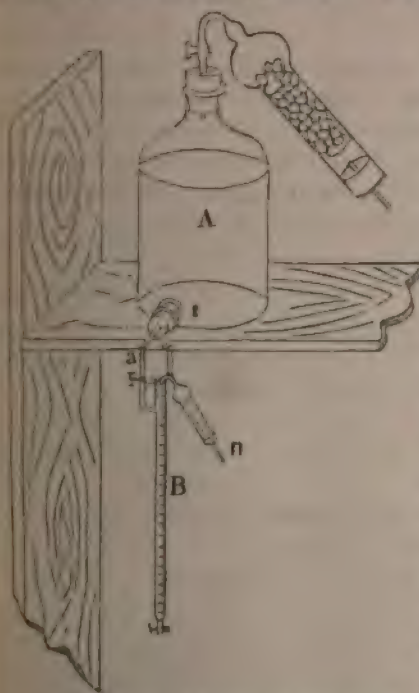
therefore first prepared a *soluble* potato starch according to Zulkowsky's directions, and mixed its watery solution with excess of baryta water. After the precipitate had settled I syphoned off the supernatant fluid, and washed the deposit with alcohol of 45 per cent., until all alkalinity was removed. The baryta starch is insoluble in excess of baryta and in weak alcohol, though slightly soluble in pure water. After collecting precipitate on a filter, and washing a few times with ether, I then dried it and made an analysis. 2.0275 grms. were dissolved in hot weak hydrochloric acid, and the liquid made up to 250 c.c. 50 c.c. were then precipitated with sulphuric acid, the precipitate treated and weighed as usual, and calculated to baric oxide. I now found the amount of base to be 19.97 per cent., which fairly well agrees with the formula—



which requires 19.10 per cent BaO.

The slight excess of BaO is due to a small admixture of barium carbonate.

Further experiments proved a similar precipitate is formed, even when the starch is merely in the state of paste, and not actually dissolved. Wheat starch, boiled with water for half an hour, yielded with baryta a compound containing 19.8 per cent. BaO.



The gravimetric estimation being too difficult, I thought the best and simplest way would be to dissolve the starch, then to add a definite amount of baryta water of known strength. To completely separate the compound I added proof spirit to a definite volume, and afterwards titrated an aliquot part with decinormal hydrochloric acid. As baryta water quickly absorbs carbonic anhydride and gets turbid, I made use of a special apparatus (see illustration), which will not need a detailed explanation. Sufficient to say the tube connected with the jar A is filled with quicklime, whilst the tube n contains small lumps of caustic potash.

Three grms. of the starch were rubbed in a mortar with 150 c.c. water, introduced in a 250 c.c. flask, and heated in waterbath for half an hour, with occasional shaking. After cooling, 50 c.c. of baryta water were added from the burette, the flask corked and well shaken for a few minutes, and finally the contents diluted up to the mark with proof spirit.

After ten minutes, it was found easy to pipett off 50 c.c. of the clear fluid, and to titrate the excess of BaO with decinormal hydrochloric acid, using phenolphthalein as indicator. The interval during the settling of precipitate I utilised in standardising the baryta water.

The difference in BaO between the two determinations was first multiplied by 5 then by 4.32 = amount of starch. The following test analyses show the accuracy of the method:—

PURE WHEAT STARCH.			
Moisture.	Ash.	Starch (by difference).	Starch (found).
15.03—15.07	.08	84.89—84.85	84.32—84.52—84.65
MAIZE STARCH.			
Moisture.	Ash.	Starch (by difference).	Starch (found).
13.83	.06	86.11	85.65—85.85
POTATO STARCH.			
Moisture.	Ash.	Starch (by difference).	Starch (found).
18.03	.28	81.72	78.7—78.57

The deficiency in starch (about 3 per cent.) is partially accounted for by impurities. This sample did not dissolve clear in acids.

I now proceed to give the details of the process when applied to cereals. In every case I have made a full analysis of the other constituents. The samples must be finely ground in a coffee-mill.

#### ESTIMATION OF THE STARCH.

Weigh out about three grms. of the flour, and rub well in a mortar with cold water; but in the case of hard substances like maize, rice, peas, boiling water must be used. Pour off into a 250 c.c. flask, and rub residue again and again with small quantities of water, until the bulk of the starch is removed; finally introduce residue also into the flask. Add water up to about 100 c.c., and heat in waterbath for half an hour, with occasional stirring. After cooling, add 50 c.c. of standard baryta water; cork the flask, and shake well for two minutes. To completely separate the baryta starch, add proof spirit up to the mark. After ten minutes pipette off 50 c.c., but if a flocculent precipitate refuses to settle, filter a portion of the fluid through glasswool contained in a tube. The titration and calculation have already been described. I use 10 c.c. baryta water, and add 50 c.c. of thoroughly boiled distilled water before checking it.

An analysis can be finished in about an hour and a half. I must call attention to the fact that if the liquid is not frequently shaken during the heating, there will be a tendency to form lumps of starch paste, which are but imperfectly acted on by baryta water. The following results have been obtained with various cereals\* :—

##### 1. MAIZE.

(a) Used 3 grms., 50 c.c. = 19.5 c.c. acid; 10 c.c. baryta water = 32.1 c.c. acid. Difference 12.6 c.c. acid = 68.04 per cent. starch.

(b) Used 3 grms. filtrate = 19.5 c.c. acid = 68.04 per cent. starch.

##### 2. BUCKWHEAT (THE HUSK REMOVED).

(a) Used 3 grms., 50 c.c. filtrate = 20 c.c. acid; 10 c.c. baryta water = 32.3 c.c. acid. Difference 12.3 c.c. acid = 66.42 per cent. starch.

(b) Filtrate = 19.9 c.c. acid = 66.96 per cent. starch.

(c) Same as b.

##### 3. BARLEY.

(a) Used 2.75 grms. filtrate = 19 c.c. acid; 10 c.c. baryta water = 30.70 c.c. acid. Difference 11.7 c.c. acid = 69.68 per cent. starch.

(b) Used 2.018 grms. filtrate = 22 c.c. acid = 69.83 per cent. starch.

(c) Used 2.34 grms. filtrate = 20.8 c.c. acid; 10 c.c. baryta water = 30.8 c.c. acid = 69.22 per cent. starch.

\* If dextrine is present, this is also precipitated, and will count as starch.



## 4. WHEAT FLOUR.

- (a) Used 2.936 grms. (by accident *a, b, c*, only mixed with 49.8 c.c. baryta water), filtrate = 17.08 c.c. acid, 10 c.c. baryta water = 30.4 c.c. acid = 73.49 per cent. starch.  
 (b) Used 2.903 grm., filtrate = 17.23 c.c. acid = 73.52 per cent. starch.  
 (c) Used 2.97 grm., filtrate = 16.92 c.c. acid = 73.52 per cent. starch.  
 (d) Used 3.032 grm., filtrate = 16.7 c.c. acid = 73.22 starch.

## 5. PEAS.

- (a) Used 3.212 grm., filtrate = 19.05 c.c. acid, 10 c.c. baryta water = 30.4 c.c. acid = 57.24 per cent. starch.  
 (b) Used 2.969 grms., filtrate = 19.95 c.c. acid, 10 c.c. baryta water = 30.35 c.c. acid = 57.29 per cent. starch.

I now proceed to give in detail the methods used in estimating the other constituents.

MOISTURE.—Weigh in a platinum dish about 5 grms. of the flour, and dry at 105°C till constant weight.

ASH.—Now incinerate, and burn to white ash.

TOTAL ALBUMINOIDS.—Treat 1 grm. of the flour according to my modification of Kjeldhal's process (see ANALYST, May, 1886). Multiply percentage of nitrogen by 100  
15.5

FAT.—Put 10 grms. of the flour into a filter-paper cartridge, and extract, in a Soxhlet's apparatus, with pure ether.

FIBRE.—Boil 3 grms. with a mixture of 150 c.c. water, and 50 c.c. 5 per cent. sulphuric acid for half an hour. Filter by aid of filter-pump, wash once or twice with water, rinse precipitate back into the beaker, and boil with a mixture of 150 c.c. water, and 50 c.c. of 5 per cent. caustic potash, for another half an hour. Finally collect residue on a weighed filter, wash first with water, then with spirit, then with ether. Dry till constant weight is obtained. As it always contains some mineral matter, incinerate, and weigh the ash.

## FULL ANALYSIS OF SOME OF THE MORE IMPORTANT CEREALS.

Name.	Moisture.	Ash.	Albuminoids.	Fibre.	Fat.	Starch.	Total.
Maize	12.68	1.78	9.63	1.97	6.12	68.04	100.22 per cent.
"	—	—	9.58-9.69	—	—	68.04	
Buckwheat (the husk removed)	13.23	1.85	13.51	2.24	2.46	66.78	100.07 per cent.
"	—	—	—	—	—	66.96	
"	—	—	—	—	—	66.43	
"	—	—	—	—	—	66.96	
Barley	13.07	2.65	9.94	2.67	1.99	69.58	99.90 per cent.
"	—	—	—	—	—	69.83	
"	—	—	—	—	—	69.23	
"	—	—	—	—	—	69.86	
Wheat flour	9.56	1.77	12.51	1.04	1.63	73.43	99.94 per cent.
"	—	1.78	—	1.09	—	73.49	
"	—	—	—	1.02	—	73.49	
"	—	—	—	—	—	73.52	
"	—	—	—	—	—	73.22	
Peas	11.47	2.34	22.34	5.57	1.12	57.26	100.1 per cent.
"	—	—	—	—	—	57.24	
"	—	—	—	—	—	57.29	

It will be noticed, my analyses of these cereals rather differ from the published ones, in so far as there is no room for sugar, which is supposed to be present in perceptible quantities, sometimes as much as 4 per cent. Sugar is certainly not precipitated by baryta water, not even if proof spirit is added. I have also never been able to detect it in the filtrate, but in peas I have found an *albuminous* body which, after boiling with hydrochloric acid, acquired the property of reducing Fehling's solution.

[NOTE BY TRANSLATOR. No doubt this process will work well with such bodies as mixed mustard, pepper, etc., if they are first washed with alcohol and ether, to remove resinous and fatty matters.—L. DE K.]

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

STARCH IN MUSTARD.—In the American Record of Pharmacy, H. Trimble suggests the amounts of ash found in ground mustard as a guide to the amount of starch found previously by the microscope. Experiments are given by R. C. Werner, who finds the normal ash to be 6 per cent., but it is not stated whether this figure applies to the mustard previously dried at 212° or not. The experiments are only 5 in number and the idea wants confirmation because it is evident that the ash in any commercial sample depends a good deal upon the degree of refinement from husk, etc. W. H. D.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

A TEST FOR MORPHINE. G. VULPIUS. Arch. d. Pharm., 225—256.—The author substitutes sodic phosphate for potassic arseniate. If a little morphine, not less than .00025 gram., is first moistened with six drops of sulphuric acid, then mixed with a few centigrammes of sodic phosphate, and now heated, until white fumes appear, the colour first becomes violet, afterwards brown. If after cooling a few drops of water are added, the colour turns a fine red, but the addition of about 5 c.c. of water makes it dirty green. If the liquid is now put into a test tube, and shaken with an equal volume of chloroform, the latter will, after subsiding, be found to be of a fine blue colour. The blue colour obtained in the well known test with morphine and neutral ferric chloride is not soluble in chloroform. L. DE K.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

SEPARATION OF GOLD AND PLATINUM FROM ANTIMONY, ARSENIC, AND TIN. DIRVELL. Dingl. pol. Journ. 263, 538.—The sulphides, precipitated by hydrochloric acid from their solution in ammoniac sulphide, are dissolved in nitro-hydrochloric acid, and mixed with a small quantity of a saturated solution of sodic oxalate. A solution of oxalic acid is now added, and then an excess of pure sodic hydrate. If sodic oxalate separates out this must be removed by filtration. The liquid is now heated to 100° C., and the gold and platinum precipitated by a small excess of chloral hydrate. The filtrate is diluted with water, and boiled for some time to remove the chloral, then acidified with hydrochloric acid. Excess of sulphurous acid is now added, and the antimony precipitated hot by sodic bisulphite and sodic hyposulphite. The filtrate is mixed with large excess of hydrochloric acid, and precipitated hot with hydric sulphide, to get the arsenic. The large amount of acid prevents precipitation of the tin, which is then estimated as usual. L. DE K.



# THE ANALYST.

AUGUST, 1887.

## PROGRESS OF THE BUTTER-SUBSTITUTES BILL.

THE following is from the *Times* Parliamentary report of 9th July:—

The House went into Committee on this Bill.

Mr. KENNY moved to substitute the word "oleomargarine" for the word "butterine."

Mr. JACOB BRIGHT moved to report progress.

The motion was negatived.

Mr. ILLINGWORTH objected to giving an article of commerce a bad name if it could be avoided.

Mr. BIGGAR said that the bad name would be applied to an article made from lard, worth 4d. a pound, and sold as butter at the price of butter.

Mr. JACOB BRIGHT said the evidence before the Committee was that butterine was a wholesome article, with which bad butter could not compete.

Sir R. PAGET, having originally opposed the name butterine, would now retain it, and thought that the Bill in its present state would sufficiently carry out the object which they had in view.

Mr. A. O'CONNOR, as representing an agricultural constituency, supported the amendment.

Mr. RITCHIE opposed the amendment as unnecessary for the protection of purchasers.

Dr. CLARK supported the adoption of the word "margarine" without "oleo." It was necessary to distinguish between that which came from the living cow, and that which was made from the cow when it was dead, and butterine did not sufficiently indicate the difference.

Mr. ADDISON disputed the right of Parliament to fix names for articles of commerce.

Mr. KENNY said that Parliament legislated against fraudulent trade-marks, and the name butterine had been adopted by the trade because its similarity to butter facilitated the deception of the public.

The Committee divided, and the numbers were—

For the amendment	..	..	..	..	..	87
Against	..	..	..	..	..	70
Majority	..	..	..	..	..	—17

The word "butterine" was, therefore, struck out. It was agreed to amend the amendment by substituting "margarine" for "oleomargarine," and, after a short conversation, the word "margarine" was inserted. This involved several consequential amendments by the substitution of "margarine" for "butterine."

Mr. JACOB BRIGHT moved to amend the penalties clause by taking away the option of imprisonment.

Mr. KENNY said the Committee on the Bill, after full consideration, determined that imprisonment should be retained.

Mr. ILLINGWORTH thought the money penalties would be sufficiently deterrent.

Mr. MACLURE opposed imprisonment.

Mr. F. S. POWELL said there was a tendency in modern legislation to form new crimes. (Ironical cheers.) The clause put too much power in the hands of magistrates. (Ironical cheers.) Under the Adulteration Acts imprisonment could not be ordered unless the adulterant were injurious to health.

After further conversation the amendment was agreed to, and the penalty of imprisonment was struck out.

The provision that small quantities should be labelled "butterine, not butter" was altered to "margarine," the words "not butter" being struck out.

Mr. ADDISON moved the omission of Clause 4, embodying the description of the label to be used by retailers, which he criticised as needlessly minute.

Several members said the description had been accepted by representatives of the trade, and others replied that the change of name from butterine to margarine had rendered unnecessary the stringent conditions that were accepted with a less objectionable name.

Sir R. PAGET said the clause was essential to the Bill, and was really framed on the practice and suggestions of traders.

The Committee divided, and the numbers were—

For the clause .. .. .	106
Against .. .. .	23
Majority .. .. .	—83

The clause was therefore retained.

Mr. ADDISON move the omission of Clause 6, which gives the right to inspect premises.

Sir R. PAGET said the clause was inserted at the request of the largest manufacturer of margarine.

The clause was agreed to.

The Bill passed through Committee.

On 11th July, this Bill, as amended, was again considered.

Mr. MACLURE moved to amend the title of the Bill by substituting "butterine" for "margarine."

After some discussion the House divided, and the numbers were—

For the amendment .. .. .	99
Against .. .. .	124
Majority against .. .. .	—25

After certain amendments had been agreed to, the Bill was read a third time. The word margarine thus finally remains in the Bill.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

### PEPPER.

By THOMAS STEVENSON.

*Read at the Meeting, June, 1887.*

THE following determinations may be of interest. The peppers were ground by myself; and 50 grms. of pepper were operated upon in each case. The ground samples were exhausted with hot methylated spirit of wine, 60 O.P., an operation extending over several days.



The alcoholic liquids were evaporated, and the extracts digested with a cold solution of potash. The piperine thus separated was washed with water, dried at 100° C., and weighed. It was then re-crystallised from alcohol, and re-weighed in a high state of purity. The resin was obtained by precipitating the potash solution with hydrochloric acid, and was no doubt a mixture of resinous and oily bodies. The results are given in terms of air-dried pepper; and each of the samples lost about 14 per cent. of its weight when dried in the water-oven.

	Piperine per cent.	Resin per cent.
Black pepper .. .. .	7.14	1.44
" (Trang) .. .. .	6.62	0.82
White pepper .. .. .	6.47	0.69
Long pepper .. .. .	4.24	1.16

Long pepper is inferior in pungency to black pepper; but it is doubtful whether the contention that where "pepper" only is asked for, and long pepper or a mixture of black and long peppers is supplied a fraud is committed, can be maintained. The term "pepper" was at one time undoubtedly applied to both round and long peppers; and it is only within recent years that long pepper has been removed by botanists from the genus *piper*. I am clear, however, that if either "black" or "white" pepper be demanded, long pepper, or a mixture, ought not to be supplied.

#### NOTES ON THE LOGWOOD TEST FOR ALUM IN BREAD, ETC.

By W. C. YOUNG, F.I.C., F.C.S.

(Read at the Meeting, June, 1887.)

IN my paper on this subject, read at the December meeting of last year, I assumed, from the results of experiments therein described, that the reaction with the logwood test was only obtained when the alumina was present in a form soluble in water; and I stated my opinion that it was possible that under some circumstances no reaction would be obtained, even if alum had been added. In the course of the discussion it was pointed out that phosphate of alumina was insoluble in acetic acid, and that therefore my results could not be explained on the assumption that the alumina naturally present in the flour existed in the form of phosphate. To this I replied that it was possible that the alumina, in whatever form it existed, was combined with the gluten; and this compound might, like gluten, be insoluble in water, and soluble in acetic acid.

I have carried the investigation a little farther, and now beg to lay the results before you.

With reference to the insolubility of phosphate of alumina in acetic acid, I find it is not so insoluble as is supposed; for if to a weak solution of alum a drop of solution of phosphate of sodium is added, the precipitate formed is dissolved on the addition of acetic acid.

Again, no precipitate is obtained by the addition of a drop of solution of phosphate of sodium to a weak solution of alum acidified with acetic acid.

In both instances the precipitate was immediately produced on warming the liquid, but again disappeared when the liquid was set aside in the cold for a few hours.

It is therefore evident that phosphate of alumina is distinctly soluble in cold, and insoluble, or nearly so, in hot, acetic acid.

As far as I have been able to ascertain, this peculiar property of phosphate of alumina has hitherto escaped notice,\* and it will at once be seen that it must affect the accuracy of such processes for the estimation of alumina in bread, etc., as are based upon the insolubility of phosphate of alumina in acetic acid. I have a number of experiments in progress upon this point, the results of which I hope to be able to bring before the Society at an early date.

Having observed, as described in my former paper, that the addition of acetic acid to bread, which had previously given no reaction with the logwood test, had the effect of producing the blue colour in a very marked manner, I thought it possible, by soaking the bread in acetic acid, to dissolve out the alumina and leave the bread free from it; but I find by experiment that after repeated soakings (the bread being subsequently washed free from acid) the reaction is obtained on again acidifying, the successive acid solutions also giving a marked reaction with alkaline logwood solution.

It is therefore evident that the alumina naturally present is so thoroughly diffused throughout the whole substance of the bread that it is almost impossible to extract it in this way. My next experiment was directed towards ascertaining whether pure wheat starch, or gluten, or both, contained alumina sufficient to produce a reaction after treatment with acetic acid.

I found it extremely difficult to separate the last trace of gluten from the starch; but when I obtained the starch quite free from it I was unable to get the least change of colour with the logwood reagent after treatment with acetic acid. On the other hand, every sample of gluten I prepared gave a marked reaction when treated in a similar manner.

This result shows conclusively that the alumina is contained in the gluten, and is absent from the starch, and explains the difficulty experienced in endeavouring to dissolve the alumina out of bread, as above stated.

Some doubt having been expressed as to whether phosphate of alumina had the same property of fixing the logwood colour as the hydrate, I made the following experiments:—

Two portions of a solution of alum were taken, each coloured with tincture of logwood; to one a few drops of solution of ammonia were added, and to the other a few drops of solution of phosphate of soda. In both cases the colour was carried down with the precipitate.

This experiment showed that *immediately on precipitation* both had the same property of fixing the colour, and on repeating the experiment, by adding the tincture of logwood *after* the precipitation of the hydrate and phosphate of alumina, I found that both precipitates extracted the colour.

In order to ascertain whether these precipitates lost this property by keeping, I next prepared some hydrate and phosphate of alumina by precipitation, thoroughly washed the precipitates on filters, and set them aside for ten days; at the end of this time they were still in a gelatinous condition, but were apparently quite free from superfluous moisture. I then suspended a portion of each in water and added alkaline logwood solution, when I found that both still retained the property of mordaunts.

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\* Bell, in his "The Chemistry of Foods," part II., p. 113, gives a modification of Dupré's process for estimation of alum in flour, etc., and states that he has found that the whole of the alumina is not precipitated in the cold, and that the remaining alumina could be recovered on boiling.



I may here mention that the colour given with the phosphate appears much lighter than with the hydrate when seen by reflected light, which is, no doubt, due to the greater opacity of the former, as when seen by transmitted light the colours are identical.

It would seem from the results of these experiments that if the alumina in bread were present in the form of free hydrate or phosphate, a reaction would always be given with the logwood test, and further, that it is necessary to get the alumina into one of those forms in order to obtain the blue stain; therefore, I was not strictly correct in assuming in my former paper that the logwood test would fail, unless the alumina in the bread was in a soluble form, although I still believe that practically it is always the case when the reaction is given.

Another conclusion which may fairly be drawn from my results is that the alumina naturally present in flour is *combined* with the gluten. This being so, I thought it would be interesting to see if gluten would absorb alumina from a solution of alum. With this object I prepared some gluten, thoroughly dried it, and found the ash equal to 1.28 per cent. A portion of this gluten finely powdered was placed in a 2 per cent. solution of alum, and kept for some hours at a temperature of about 180°F.; it was then thrown on to a filter, thoroughly washed, and dried. The ash then amounted to 1.32 per cent. Taking into consideration the great difficulty of washing the gluten free from excess of alum, I do not think this slight increase of ash is due to absorption of alumina. Although gluten does not appear to have the property of absorbing alumina from a solution of alum, yet its properties are affected in a marked degree by contact with such a solution, as it may then be kept moist in a warm place without giving any indication of decomposition, whilst ordinary gluten, as is well known, darkens in colour, swells, and rapidly decomposes under similar circumstances.

With reference to my former statement that the logwood test may fail, even if alum had been added to the bread, I have tested a large number of samples containing alum in different quantities, and made and baked in a variety of ways, and I am bound to say that in every case I have obtained a reaction, such as could not be in any way overlooked.

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#### QUANTITATIVE DETERMINATION OF PEPPER MIXTURES.

By A. W. STOKES, F.C.S., F.I.C.

*Read at the Meeting, June, 1887.*

SEVERAL methods have been suggested for the estimation of mixtures of pepper with other substances, though none, I believe, has resulted in a quantitative formula.

Thus, Mr. W. Blyth suggests that the nitrates and nitrites might form a guide; but as he finds they vary in genuine peppers from .038 to .118 per cent. (when calculated as nitric acid) little reliance can be placed upon this for a quantitative method.

The quantity of piperine has been proposed also as an index; but in genuine peppers, including long pepper, this varies from 1.7 to 9.38 per cent. Even in a variety

of black pepper samples the piperine ranges from 4 to 9.38 per cent. Nor is the estimation of piperine an easy and rapid operation.

Mr. C. Heisch lays great stress upon the percentage of starch natural to pepper, stating that he would "view with great suspicion" a pepper containing less than 50 per cent. of starch. Yet his own analyses (*THE ANALYST*, vol. xi, p. 188) show a sample of genuine Acheen Penang containing only 48.53 per cent., while a mixture of 90 per cent. of black pepper with 10 of Poivrete is shown to contain 49.98 per cent. of starch. In nine samples of black pepper, Mr. Heisch's analyses show a variation from 48.5 to 56.6 per cent. of starch.

In two samples of long pepper the starch is given as 46.1 and 58.9 per cent. Pepper-starch cannot be rapidly or easily estimated.

For these reasons the quantity of starch found in a mixture would not well serve for quantitative purposes in fixing the amount of adulteration.

The amount of ash is sufficient to show any abnormal proportion of sand and dirt; but for admixture with olive-stones, rice, and starch generally, no quantitative method is known.

In my opinion the determination of the woody-fibre will, as a rule, be sufficient for obtaining a very close approximation to the extent of admixture. In separating woody-fibre by the usual method of boiling the substance, first with acid, and then with alkali, constant results are difficult to obtain. The alkali always dissolves up part of the fibre; how much, depends upon the fineness to which the substance is ground. From pepper mixtures, to obtain the woody-fibre, we have to get rid of starch, ash, piperine, and resinous bodies. This I find can best be done by taking, say 1 gm. of the substance, boiling it in a flask for five minutes with 100 c.c. of distilled water. This swells out the starch granules. Now 50 c.c. of distilled water containing 6 c.c. of strong sulphuric acid are added, and the whole is boiled for one hour under an upright condenser. The contents of the flask are then washed on to a weighed filter-paper, carefully washed with hot distilled water, then with hot alcohol, and lastly with ether. The filter-paper is placed in a weighing tube, dried, and weighed. Some allowance should be made for loss of weight of the filter-paper when extracted by hot sulphuric acid solution, alcohol, and ether. Filter-papers (English) of  $5\frac{1}{4}$  inches diameter lost .012 gramme when submitted to this process. It is then incinerated, and the weight of the ash subtracted from the former weight. Thus determined, closely concordant percentages of fibre are obtained from the same sample.

Taking a number of samples of the same peppers, etc. (for some of which I am indebted to the kindness of Dr. T. Stevenson), I find the following percentages of fibre (in the dried samples).

Black pepper 21.0 to 26.3 with an average of 24.4 per cent.				
White	"	12.7 to 13.8	"	" 13.3 "
Long	"	20.0 to 22.3	"	" 21.0 "
Olive stones		62.2 to 64.2	"	" 62.5 "
Rice		0.8 to 1.6	"	" 1.0 "

A more extended series of analyses might perhaps alter these averages slightly, but not, I think, materially.



Accepting these averages as correct a general formula for calculating a mixture of olive stones with pepper would be

$$x = \frac{100a - 100m}{a - p}$$

Where  $x$  = percentage of dry pepper.

$a$  = " " fibre in dry olive stones.

$m$  = " " " mixture.

$p$  = " " " pepper.

The formula for a mixture of black pepper and olive-stones would be for instance :—  $x = \frac{6250 - 100m}{38.1}$

Mixtures of pepper with the above adulterants were made, and the fibre determined.

A mixture of black pepper and olive stones containing :—

10 per cent. olivestones gave 28.6 per cent. fibre, using formula gives 11.1 per cent. olive stones.

20 " " 33.8 " " " 24.7 " "

30 " " 36.6 " " " 32.1 " "

A mixture of white pepper and olive stones containing :—

10 per cent. olivestones gave 18.4 per cent. fibre, this formula gives 10.4 per cent. olive stones.

20 " " 23.5 " " " 20.8 " "

30 " " 28.7 " " " 31.3 " "

A mixture of long pepper and olive stones containing :—

10 per cent. olivestones gave 24.9 per cent. fibre, this formula gives 9.4 per cent. olive stones.

20 " " 29.0 " " " 19.3 " "

30 " " 33.3 " " " 32.1 " "

A mixture of rice with white pepper containing :—

10 per cent. rice gave 11.9 per cent. of fibre, this by formula gives 11.3 per cent. of rice,

20 " " 11.0 " " " 18.7 " "

30 " " 9.8 " " " 28.4 " "

Rice is usually added only to white peppers, so that the formula in such case would be Rice, per cent.  $= \frac{1330 - 100m}{12.3}$

In the case of mixtures of white and black peppers to which olive stones have been added, it is necessary to estimate by the appearance and by the ash the probable proportions of white and black present, and then to take as the percentage of fibre naturally present some number between 13.3 and 24.4 for the formula in calculating results. The result will necessarily be only approximate.

Of course the presence of olive stones and of starch (foreign) should first be discovered by the microscope before applying the quantitative method indicated.

For mixtures of long pepper with other peppers the process is not applicable. I would here express my thanks to my assistant, Mr. E. Michael, for care and attention in carrying out much of the above work.

*Conclusion of the Society's Proceedings.*

## METHOD OF SEPARATION OF COLOURING MATTERS IN BUTTER, IMITATION BUTTER, AND SO-CALLED BUTTER COLOURS.

BY PROF. ALBERT R. LEEDS, PH.D.,

*Professor of Chemistry in Stevens Institute, Hoboken, N. J.*

IN the case of butter and imitation butter which contain minute quantities of colouring matters, 100 grams. of the sample are dissolved in 300 c.cm. of petroleum ether. The specific gravity of this ether, which I have myself used, has been 0.638. It evaporates completely, leaving no residue or trace of colour behind. A graduated cylinder or "test-mixer" can be advantageously used in making the solution. The ethereal solution of the fats and colouring matters is separated from the water and salts by means of a separating funnel. It is washed with 100 c.cm. of water in successive portions, and these wash-waters are drawn off by the separating funnel. The ethereal solutions of the fat are allowed to stand, in winter, in the cold, or surrounded by ice-cold water, for 15 or 20 hours, when a large quantity of stearin crystallises out. In some cases the stearin thus separated amounted to 20 per cent. of the total weight.

The clear yellow ethereal solution, after being decanted from the separated stearin, is shaken up in the test-mixer with 50 c.cm. of a decinormal solution of potash. This is usually sufficient to dissolve out all the colouring matters which are capable of being dissolved by dilute alkali.

After the aqueous solution of the colouring matters has been drawn off from the ethereal solution of the fats, this aqueous solution is very carefully acidified with dilute hydrochloric acid until just acid to test paper. The colouring matter (accompanied by a small amount of fatty acid which unfortunately is always formed by the saponification of the fats and subsequent setting free by the acid) then separates out. It is filtered through a tared filter and washed with cold water.

In one experiment, in which only 50 grams. of oleomargarine were thus treated, the colouring matter and some fatty acid accompanying it weighed 0.145 gram.

It is important to note that in all cases which I have encountered the ethereal solution of the fats had a pale yellow colour. This slight colour was left after the first separation of the ethereal solution of the fats from the aqueous solution of the colouring matters in dilute alkali. It was not removed or lessened by any subsequent treatment with potash solution, however oft repeated. This residual pale yellow colour is due to the fats and oils themselves, and it is not due to any added colouring matters. This remark is true of butter, imitation butter, and "oleo oil." It is the slight yellow colour natural to the fat itself. It may be entirely removed without the use of chemical agents, and without any change whatsoever in the fat itself, except the abstraction of that very minute portion which carries with it the yellow colour.

### BUTTER COLOURS.

Three samples of butter colour were similarly treated, using, of course, smaller quantities of the samples. About 5 grams. were dissolved in 20-25 c.cm. petroleum ether, and 10 c.cm. of a 4 per cent. solution of potash were used for separation. The aqueous solution in alkali was then just acidified with dilute hydrochloric acid, and the colouring matter filtered and washed on a tared filter. It was then left behind in a resinous condition.



## REACTIONS OF COLOURING MATTERS.

Colouring Matter.	Concentrated $H_2SO_4$	Concentrated $HNO_3$	$H_2SO_4$ + $HNO_3$	Concentrated $HCl$
Annatto.	Indigo Blue, changing to Violet.	Blue, becoming colourless on standing.	Same.	No change, or only slight Dirty Yellow and Brown.
+ Annatto decolourised butter.	Blue, becoming Green, and slowly changing to Violet.	Blue, through Green and Bleached.	Decolourised.	No change, or only slight Dirty Yellow.
Turmeric.	Pure Violet.	Violet.	Violet.	Violet, changing to original colour on evaporation of $HCl$ .
Turmeric + decolourised butter.	Violet to Purple.	Violet to Reddish-Violet.	Same.	Very fine Violet.
Saffron.	Violet to Cobalt Blue, changing to Reddish-brown.	Light Blue, changing to light Reddish-Brown.	Same.	Yellow, changing to Dirty Yellow.
Saffron + decolourised butter.	Dark Blue, changing quickly to Reddish-brown.	Blue, through Green to Brown.	Blue, quickly changing to Pale.	Yellow becoming Dirty Yellow.
Carrot.	Umber Brown.	Decolourised.	Do. with $NO_3$ fumes and odour of burnt sugar.	No change.
Carrot + decolourised butter.	Reddish-brown to Purple, similar to Turmeric.	Yellow and decolourised.	Same.	Slightly Brown.
Marigold.	Dark Olive-Green, permanent.	Blue, changing instantly to Dirty Yellow-Green.	Green.	Green to Yellowish-Green.
Safflower.	Light Brown.	Partially decolourised.	Decolourised.	No change.
Aniline Yellow.	Yellow.	Yellow.	Yellow.	Yellow.
Martius Yellow.	Pale Yellow.	Yellow, Reddish precipitate. Magenta at margin.	Yellow.	Yellow precipitate, treated with $NH_3$ and ignited, deflagrates.
Victoria Yellow.	Partially decolourised.	Same.	Same.	Same colour returns on neutralising with $NH_3$ .

All were solutions in Alcohol. Solutions and reagents were used in equal proportions, two to three drops of each. Ammonia gave with Turmeric Reddish-Brown, returning to original colour on driving off  $NH_3$ .

## NITRATES AND NITRITES (II).

By A. PERCY SMITH, F.I.C., RUGBY.

In my previous paper on this subject (ANALYST, March, 1887) I showed only that a fair agreement existed between the total nitrogen and the conjoined estimations of nitrates and nitrites. My present aim is to prove that the method gives absolutely accurate results.

In my early experiments, the results were far from uniform, owing to the extraordinary delicacy of the naphthylamine test. The source of error lay in the water used. It was necessary to prepare water absolutely free from nitrogen. To do this it is best to treat it with a copper-zinc couple for *at least* a week, render alkaline, and distil slowly, rejecting that portion containing ammonia. Ordinary distilled water gives a strong colouration with the naphthylamine. I believe the nitrous acid is chiefly the result of oxidation of the ammonia, which all distilled water contains.

After eliminating all sources of error, I found that the determination of nitrates and nitrites when known quantities were added to *pure* water agreed absolutely with the colours produced for comparison by known quantities of the salts used separately. For instance: A litre of nitrogen-free water has added to it 5.0 mg. N in the form of  $\text{KNO}_3$ , and .3 mg. N as  $\text{KNO}_2$ .

100 c.c. is evaporated to dryness for nitrates, and the colour produced by the phenolsulphonic acid test is identical with that yielded by .5 mg. N similarly treated. Hence 1 litre = 5.0 mg.

Again, 100 c.c. are tested for nitrites, and the colour is copied by .03 mg. N = .3 mg. per litre.

In applying the naphthylamine test, it is necessary that the solution of naphthylamine hydrochlorate should be colourless, or nearly so. The colouration developed by standing is easily removed by animal charcoal. It is a good plan to keep charcoal in the solution, the supernatant liquid can be taken up by a small pipette, from which the drop or drops is added to the water under examination.

Similar quantities of the reagents must be used in each tube. This is especially necessary in determining nitrates. Different *shades* (not depths) of colour may be produced if attention is not paid to this point. I find the most trustworthy method is to evaporate *just* to dryness, or just short of dryness, add ten drops phenolsulphonic acid from a small (1 c.c.) pipette, agitate, add 1 c.c. water, and three drops strong sulphuric acid, then dilute, add ammonia in excess, and make up to 100 c.c.

The naphthylamine test is far superior to metaphenylene diamine for ascertaining when all nitrites are reduced by the copper-zinc couple, and its use has led me to the opinion that the surface of zinc recommended by the water committee, viz.,  $3 \times 2$  inches, is insufficient for the purpose; that is, if one desires the reduction to be completed in twenty-hours. Using that amount of zinc in 100 c.c. of water of  $42^\circ$  of hardness led to the following:—

					N per litre.	Naph. test.
In 24 hours	..	..	..	..	1.03	Red
" 3 days	..	..	..	..	1.71	"
" 4 "	..	..	..	..	2.04	"

It was then kept at  $80^\circ\text{F.}$  for a whole day.



In 7 days.. .. . 3.71 None

Again. (1) 50 c.c. of standard potassic nitrate solution was made up to 500 c.c. with this same hard water; the total nitrogen expressed as ammonia being 10.1412 milligrammes.

(2) 50 c.c. of potassic nitrate solution was diluted in a similar manner with pure distilled water. Total N as  $\text{NH}_3 = 7.8787$  mg. 100 of each was treated with 3'' x 2'' copper-zinc couple.

				Soft Water.	Hard Water.
Milligrammes of $\text{NH}_3$	..	..	..	7.8787	10.1412
In 2 days	..	..	..	1.3	2.5
" 4 "	..	..	..	4.0	4.25
" 6 "	..	..	..	7.8	10.1

It would seem from this as if the hardness of the water has little or no effect upon the time required; indeed a hard water seems to be reduced the easier of the two, but the quantity of zinc exposed is no doubt the chief factor in the duration of time, as may be seen from the following experiments:—

Water containing—

Total solids	1040.0	} Total Am by CuZn 12.5 milligrammes per litre.
Free Am	0.09	
Alb. "	0.12	
N	10.23	

Three lots of 70 c.c. were treated with CuZn couples, having the respective surfaces of (a) 4 sq. in., (b) 2½ sq. in., (c) 1½ sq. in.

		(a)	(b)	(c)
In 24 hours (5 c.c.)	..	.032	.0272	.020
" 48 "	..	.0625	.0625	.035
" 72 "	..	—	—	.045

$.0625 \times 200 = 12.5$ , the total amount present.

Here we see in a very hard water complete reduction took place in two days with only 2½ sq. in. of zinc. The sample having 4 sq. in. was probably finished earlier.

From these and other results, I concluded it was better to err by using too much zinc than too little, and that eight sq. in. should be invariably employed. By so doing the time required for reduction is seldom prolonged beyond the twenty-four hours. Of course I am speaking of my own experience only. It may happen that others may obtain different results. The waters in my district are generally hard, some very much so, due to the proximity of the lias. They are also highly charged with chlorine from salt deposits in the soil. (We have an artesian well just beyond the town, which is of no use to anybody on account of the salinity of the water.) This may probably tend towards rapidity, whereas, according to Mr. Williams' original paper on this subject (ANALYST, Vol. VI., p. 36), the presence of salts with an alkaline re-action is found to retard the speed of the reduction.

## SCHEME FOR QUALITATIVE BLOWPIPE ANALYSIS.\*

BY A. J. MOSES.

In the following scheme the tests are grouped in an order favourable to rapid work, and the attempt has been to give an amount of detail that will suffice for most analyses. Substances of unusual complexity will need special treatment that the scheme does not pretend to outline, and at all times successful work will be probable only when the analyst has previously acquired a fair knowledge of the reaction of the simple oxides.

The substance may contain:—Al, NH<sub>3</sub>, Sb, As, Ba, Bi, Bo, Br, Cd, Ca, Cl, Cr, Co, Cu, F, Au, I, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Si, Ag, Na, Sr, S, Te, Sn, Ti, W, U, V, Zn.

## TESTS UPON THE UNROASTED SUBSTANCE.

1. *In narrow glass tube closed at one end.*

Heat in the flame of an alcohol lamp, at first gently, and then to redness. Note all changes.

Moisture in the upper part of tube .. .. .	H <sub>2</sub> O
Odourless gas that assists combustion .. .. .	O
Pungent gas that whitens lime water .. .. .	CO <sub>2</sub>
Odour of prussic acid .. .. .	CN
" of putrid eggs .. .. .	H <sub>2</sub> S
" that suffocates, fumes colourless .. .. .	SO <sub>2</sub>
"       "       " violet .. .. .	I
"       "       " brown .. .. .	Br
"       "       " greenish .. .. .	Cl
"       "       " etch the glass .. .. .	Fl
" of nitrous acid, fumes reddish-brown .. .. .	N <sub>2</sub> O <sub>5</sub>
" of ammonia, fumes colourless or white .. .. .	NH <sub>3</sub>
Sublimate white, fusing yellow .. .. .	PbCl <sub>2</sub>
"       " and volatile .. .. .	NH <sub>3</sub> (salts)
"       " yellow hot, infusible .. .. .	HgCl
"       "       " fusible .. .. .	HgCl <sub>2</sub>
"       " fusible, needle crystals .. .. .	Sb <sub>2</sub> O <sub>3</sub>
"       " volatile, octahedral crystals .. .. .	As <sub>2</sub> O <sub>3</sub>
"       " fusible, amorphous powder .. .. .	TeO <sub>2</sub>
" mirror-like, collects in globules .. .. .	Hg
"       " does not collect in globules .. .. .	As, Cd, Te
" red when hot, yellow cold .. .. .	S
" dark red when hot, reddish-yellow cold .. .. .	As <sub>2</sub> S <sub>3</sub>
" black, when hot, reddish-brown cold .. .. .	Sb <sub>2</sub> S <sub>3</sub>
" black, but becomes red when rubbed .. .. .	HgS
" red to black, but becomes red when rubbed .. .. .	Se
Colour of substances changes	
from white to yellow, cools yellow .. .. .	PbO
"       " yellow, cools white .. .. .	ZnO
"       " dark yellow, cools light yellow .. .. .	Bi <sub>2</sub> O <sub>3</sub>
"       " brown, cools yellow .. .. .	SnO <sub>2</sub>
"       " brown, cools brown .. .. .	CdO
" yellow or red to darker, cools green .. .. .	Cr <sub>2</sub> O <sub>3</sub>
" red to black, cools red .. .. .	Fe <sub>2</sub> O <sub>3</sub>
" blue or green to black, cools black .. .. .	CuO

\* School of Mines Quarterly.



2. *In narrow glass tube open at both ends.*

Place the assay near the lower end of the tube, and heat gently, and then strongly increasing the air current by holding the tube more and more nearly vertical.

Odour that suffocates .. .. .	$\text{SO}_2$ indicating S
" of rotten horseradish .. .. .	$\text{SeO}_2$ " Se
" of garlic .. .. .	$\text{As}_2\text{O}_3$ " As
Sublimate white volatile octahedral crystals	$\text{As}_2\text{O}_3$ " As
" " non-volatile powder ..	$\text{Sb}_2\text{O}_3$ " Sb
" " non-volatile fusible powder	$\text{TeO}_2$ " Te
" " non-volatile fusible powder	$\text{PbSO}_4$ " PbS
" grey red at distance .. ..	$\text{SeO}_2$ " Se
" yellow hot, white cold, blue in R. F.	$\text{MoO}_3$ " Mo
" brown hot, yellow cold, fusible ..	$\text{Bi}_2\text{O}_3$ " Bi
" metallic mirror .. .. .	" Hg

3. *Alone on charcoal.*

Heat with gentle O. F.

Sb.—Volatile white coat that disappears in R. F. tinging the flame green.

As.—Garlic odour and a faint white coat that disappears in R. F., tinging the flame azure blue.

Se.—Horseradish odour, and a steel grey coat that disappears in R. F., tinging the flame azure blue.

Te.—White coat with red or yellow border that disappears in R. F., tinging the flame green.

Change to gentle R. F.

Cd.—Brown coat surrounded by peacock tarnish. The coat heated in a closed tube with  $\text{Na}_2\text{S}_2\text{O}_3$  gives a bright yellow mass.

Heat strongly in both flames, and if the presence of Te is suspected place a bit of porcelain so that the fumes pass between it and the coal (or test a separate portion on a plaster tablet).

As, Sb, and Cd may give stronger tests.

Zn.—White not easily volatile coat, bright green with cobalt solution.

Sn.—White non-volatile coat close to assay, blue green with cobalt solution.

Te.—Moisten the film, which forms on the porcelain, with concentrated  $\text{H}_2\text{SO}_4$ , and heat gently; the acid will be coloured a fine red.

Dissolve any coat that forms in Ph. S., and treat on coal with R. F. and tin.

Sb or Bi.—A dark grey to black bead.

White coats may form from Mo Pb Bi or alkalis, yellow coats from Pb or Bi, brown or red coats from Cu or Mo, and the ash of the coal may be white or red. If these coats interfere with the Sb test, the Pb and Bi may be held back by adding a little boracic acid.

4. *With soda on coal.*

Make a paste with the substance, soda, water, and charcoal dust, and heat strongly with R. F.

As and Cd should be noticed here even in small amounts.

If *any* coat forms, moisten it with cobalt solution, and blow a strong blue flame on the substance.

Zn.—Bright green.

Sn.—Blue green.

Dig up the coal containing the soda, and place it upon a silver coin, moisten, crush, and heat.

S, Se, or Te.—The coin will be blackened.

If the soda effervesces during fusion, look for  $\text{SiO}_2$  or  $\text{TiO}_2$ . Examine it also for metallic particles, and note whether they are *e.g.*, malleable, magnetic, brittle, red, etc.

#### 5. With bismuth flux.

Treat the substance on coal to remove the excess of Sb, As, and Hg, by gentle O. F., and excess of Cd by gentle R. F. Mix with an equal volume of the flux, and heat gently upon a plaster tablet.

Pb.—Yellow coat of iodide at distance, yellow oxide nearer.

Bi.—Red coat of iodide at distance, yellow oxide nearer.

Bi and Pb.—Orange coat of iodide at distance.

Make a similar test on coal, first moistening the coal near the assay with cobalt solution. Blow a strong flame upon the substance.

Sn.—A bluish-green coat (which is not injured by the Pb and Bi coats).

White coats may form of KI or  $\text{K}_2\text{SO}_4$ , and at times the red iodide of Bi is fringed with the yellow of Bi.

#### 6. In matrass with soda.

Mix the substance thoroughly with soda, and heat.

Hg.—Metallic mirror that can be collected into globules.

Cd, Te, and As.—Metallic mirrors that cannot be collected into globules. Break matrass, and heat mirror for odour of  $\text{As}_2\text{O}_3$ .

$\text{NH}_3$ .—Odour of ammonia.

#### 7. In matrass with $\text{KHSO}_4$ .

Mix the substance with  $\text{KHSO}_4$ , and heat gently.

Br.—Brown fumes. Gives greenish blue flame in Ph. S. bead with  $\text{CuO}$ .

I.—Violet " " emerald-green " "

Cl.—Greenish " " azure-blue " "

$\text{N}_2\text{O}_5$ .—Brown fumes, recognisable by odour.

Fl.—The glass of matrass will be etched. If a moistened strip of Brazil wood paper be inserted at one end of an open tube, and at the other a mixture of powdered phosphorus glass and substance be fused, the paper will be made straw colour, and the glass etched, and made dull and cloudy throughout, or in patches.

#### 8. With dilute acid.

Heat substance gently with dilute  $\text{HNO}_3$  or  $\text{HCl}$ .

$\text{CO}_2$ .—Effervescence continuing after flame is removed ( $\text{H}_2\text{S}$  and Cl are sometimes evolved with  $\text{HCl}$ , but may be recognised by their odours).



## TESTS UPON THE ROASTED SUBSTANCE.

If the substance has been proved to contain no As, Sb, or S, proceed with No. 9, but if not, submit the substance to a slow heating in the O. F. to drive off S, As, Sb, etc., and thus leave the metals mainly as oxides. Turn the material occasionally, and if it agglomerates, repulverise, and again roast. Continue till no noticeable fumes are given off.

9. *In phosphorus salt bead.*

Note all the changes with small and larger amounts of the substance dissolved, and with treatment in both O. F. and R. F.

The changes and their simpler confirmations are as follows:—

	In R. F. Cold.	In O. F. Hot.	In O. F. Cold.	Confirmation Soda bead O. F.
Mn.	Colourless	Brown	Violet	Bluish-Green
Cr.	Green	Reddish	Green	Yellow
Ur.	"	Yellow	"	Brown
Mo.	"	Greenish	Colourless	White
V.	"	Dark Yellow	Light Yellow	"
Co + Fe	Dull Green	Green	Green	"
Co.	Blue	Blue	Blue	On coal with Sn
W.	"	Yellow	Colourless	Green
Ti.	Violet	"	"	Violet
Ni.	Yellow	Brown	Yellow	Colourless
Fe.	Brown or Red	Dark Yellow	Light Yellow	Green to Colourless
Fe + Ur.	"	Red to Yellow	Greenish	Green
Fe + W.	"	"	Light Yellow	Blue to Wine Colour
Fe + Ti.	"	"	"	Violet
Cu.	Opaque Red or Green	Green	Blue	Opaque Red

SiO<sub>2</sub>.—The hot bead cannot be made perfectly clear, but is semi-transparent.

Touch hot bead with NaNO<sub>2</sub>.

Mn.—Violet colour.

If Cu or Co obscure the tests treat as in No. 11.

The absence of Mn and Ni is not proved until special tests have been made.

The SiO<sub>2</sub> test is best made upon a small fragment of the substance. The bases of most silicates dissolve, leaving a translucent mass or "skeleton" of the general shape of the fragment, and visible in both the hot and cold beads.

10. *Reduction colour tests.*

Saturate two Ph. S. beads with the substance in O. F., treat one of them on coal with Sn, and strong R. F., pulverise and dissolve separately in cold dilute (1-4) HCl with the addition of a little Sn. Let the solutions stand for some time, and then heat them to boiling.

	The Oxidised Bead Yields		The Reduced Bead Yields	
	In Cold Solution.	In Hot Solution.	In Cold Solution.	In Hot Solution.
W.	Blue	Deep Blue	Deep Blue	Deep Blue
Mo.	Green to Blue	Wine Brown	Brown	Faint Brown with Black precipitate
Ti.	Faint Violet	Violet and turbid	Violet and turbid	Violet
V.	Bluish Green	Green	Green	Green
Cr.	Green	"	"	"
Ur.	"	"	"	"

11. *Separation of Ni, Co, and Cu.*

If the absence of Ni not proved, or Co obscures the tests, dissolve the substance in borax on coal to saturation, and treat for five minutes in hot R. F., by which the Cu, Ni, and some of the Co will be reduced. If much Pb or Bi is present, the reduced button will be large enough to handle, but if not, add either a small (50 m.g.) gold button with a little lead, or, if much Cu is present, add only lead.

Separate the button and the slag (saving both) and scorify the button with  $\text{BO}_3$  to remove the lead, and then with frequently changed Ph. S. The metals which have united with the gold or lead will be successively oxidised, and their oxides will colour the Ph. S. in the following order:—

Co.—Blue, hot; blue, cold. May stay in the slag.

Ni.—Brown, hot; yellow, cold. May give green with Co or Cu.

Cu.—Green, hot; blue, cold. Made opaque red by Sn and R. F.

The slag should contain the more easily oxidisable metals, and be free from Cu, Ni, and Ag. Test a portion with Ph. S. and Sn to prove absence of Cu. If present it must be removed by further reduction with Pb. Pulverise the slags, and dissolve a portion in Ph. S., and treat as in No. 9.

12. *Fusion for silver.*

Fuse with test lead and borax glass. Continue the blast about five minutes, using R. F. Separate slag and button, and scorify the button with fresh borax. Transfer the button to a cupel, and blow a gentle oxidising flame across it. If the litharge is dark or the button freezes while still large, scorify again with borax, and add more lead until there remains only—

Ag.—A bright, *spherical* button, not altered by further blowing. (If the button is flat, add test lead and again cupel.)

Au.—A residue after boiling the Ag in  $\text{HNO}_3$ .

13. *Substance with cobalt solution.*

Heat the substance strongly on coal; cool, and moisten with cobalt solution.

Al. Pink, reheated, is blue.

Mg. Pink to blue, reheated, is pink.

Ba. " " brown.

Ca. Blue to green, " grey.

$\text{SiO}_2$ . " " faint blue.

Sr. Blue to brown, " grey.

Violet may be  $\text{Mg}_2\text{P}_2\text{O}_7$ . There may also be blues from fusible compounds, and greens from  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{SnO}_2$ .

14. *Flame tests.*

Moisten a clean platinum wire with HCl, dip it in the substance and heat in the blue flame, both at the tip and near the wick. Note the colour of the flame with the naked eye and through a cobalt blue glass.

	Alone.	Through blue glass.
Na.	Yellow.	Invisible.
Ba, Mo, and B.	Yellow-green.	Bluish-green.
Ca.	Yellow-red.	Greenish-grey.
Sr and Li.	Crimson.	Violet.
K.	Violet.	Purple-red.
Cu, As, Pb, Se.	Blue.	Blue.



Make similar test with  $H_2SO_4$ . The improved tests will be—

Bo.—Yellow-green.

P.—Instantaneous blue-green, that may be overpowered by the green of Bo. In such a case place a piece of Mg wire in a closed tube, and cover the wire with a mixture of soda and the substance. Heat till the mass fuses; cool, and add water.

P.—Evolution of phosphoretted hydrogen.

Make a paste of boracic acid flux and substance, and treat at tip of the blue flame.

Just after the water is driven off there may be—

Bo.—Yellow-green.

Li.—Pink to carmine.

Dissolve substance in Ph. S. to excess; add NaCl, and heat in flame gently.

Cu.—Azure blue flame.

Pb.—Azure blue flame at very high heat.

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#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

TESTING FOR BORON IN MILK, AND SIMILAR FLUIDS. M. KRETZSCHMAR. Chem. Zeit. XI. (No. 32), p. 476.—The author overcomes the difficulties in detecting the presence of boron in milk by means of the flame reaction, by employing the following method of procedure which is capable of rapid execution. The milk is well-shaken up (as any calcium borate present, is liable to settle); 5 to 6 c.c. are then transferred to a flat dish, and evaporated down to about  $\frac{1}{3}$  of the original volume. 5 to 6 drops of fuming hydrochloric acid are then added, and the evaporation continued, whilst a non-luminous flame of a Bunsen burner is directed across the dish. If any appreciable quantity of boron is present the flame will be tinged with green; in fact, the volatilization is so complete that the green colour has frequently disappeared before the other volatile constituents of the milk begin to affect the flame. It is of course impossible to tell by this means whether the boron is present as boric acid or a borate. F. W. T. K.

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#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

THE ASSAY OF OPIUM. BY E. DIETRICH. Der Pharmaceut.—The author says that he is convinced that reliable methods are known to manufacturers, but are kept secret because they constitute parts of manufacturing processes. He has tested Flückiger's method under many various conditions, to detect its defects. These were found to be: The addition of alcohol. Geissler has already demonstrated that it prevents the formation of morphine and aids separation of lime salts. The duration and intensity of shaking exerts an influence on morphine separation. When ammonia is added to morphine solution a flocculent precipitate precedes the crystalline precipitate. Examination showed the former to be narcotine. It was found when opium solution is neutralized by ammonia, narcotine only is precipitated; this can be filtered off, and when more ammonia is added, morphine separates. These facts induced Dietrich to work out a method for

which he claims the following advantages : Time of examination is shortened ; simplicity of manipulation ; no shaking ; separation of constituents ; constant results, and complete extraction of morphine.

These points are illustrated by a large array of figures given in a table.

For practical use, directions are given under three heads, viz. :

1. *For Opium.*—6 grms. air-dry opium is macerated—shaking at intervals—with 60 grms. distilled water, for twelve hours, and filtered ; to 50 grms. of the filtrate 2 cubic centimetres normal ammonia is added and filtered through a star filter of 10 centimetres diameter ; 44.2 grms. of the filtrate (=4 grms. of opium) is mixed with 10 grms. ether, in an accurately weighed Erlenmeyer wide-mouthed flask, rotated for one minute until the solution is well saturated with ether ; 4 cubic centimetres normal ammonia is now added, rotated until mixed, and placed aside for six hours at an ordinary temperature. The ethereal layer is separated and filtered through a filter of 8 centimetres diameter ; 10 grms. ether is again mixed with the opium solution in the flask, rotated for a few moments, and the ethereal layer filtered. The aqueous solution is then filtered without detaching crystals from walls of flask, and the flask and crystals are twice successively washed, each time with 5 cubic centimetres of water which has been saturated with ether. Flask and filter are dried at 100° C. Contents of filter are put into flask by means of a hair pencil, and dried until the weight remains constant. The morphine in well-developed crystals can be removed from paper without loss.

2.—*Examination of Opium Extract.*—3 grms. of extract is dissolved in 42 grms. water, placed aside one hour, 2 cubic centimetres of normal ammonia added and filtered rapidly through a filter of 8 centimetres diameter ; 31.7 grms. filtrate (= 2 grms. extract) is then treated as given under previous heading.

3. *Examination of Tincture Opium.*—50 grms. tincture of opium is evaporated one-half, and sufficient distilled water added to restore the original weight ; 2 cubic centimetres normal ammonia is added and filtered as above. 41.6 grms. (= 40 grms. tincture of opium) of the filtrate is treated as given under heading No. 1. W. H. D.

TEST FOR SULPHURED HOPS.—C.B. Gaz., Vol. XI, p. iii. If steam is conducted through hops they give up their hop oil when it condenses. If such oil be obtained from sulphured hops, when phosphoric or any other acid is added, a strong odour of sulphuretted hydrogen is developed, the presence of which can be promptly ascertained by means of lead salt. This simple test enables us to distinguish sulphured from unsulphured hops with certainty, the oil from unsulphured hops showing no such reaction.

W. H. D.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ESTIMATION OF MINUTE PROPORTIONS OF IRON IN THE PRESENCE OF ALUM OR SULPHATE OF ALUMINIUM. BY R. R. TATLOCK, F.R.S.E. Condensed from the author's original Memoir, presented to the Glasgow Section of the S.C.I.—There are two methods commonly in use for the detection and estimation of very small proportions of iron. One of these is based upon the depth of blue or green colour produced by



addition of potassium ferrocyanide, and the other upon the intensity of the red colour obtained by addition of solution of an alkaline thiocyanate to acidified solution of the substance under examination. The application of ferrocyanide for the determination of iron is not at all desirable, for various reasons. The tint is very variable—between a bright green and a full blue, even for the same proportion of iron, even when the other conditions are as nearly as possible equal, and comparison of intensity is thereby rendered unreliable; there is often a difficulty in keeping the prussian blue formed in perfect solution, even when oxalic acid is added; the composition of the blue produced cannot always be relied upon as uniform; and, lastly, it is not desirable to employ a reagent of which iron forms an essential constituent. On these grounds the use of this reagent for quantitative purposes is not to be recommended. The employment of thiocyanate of potassium ( $\text{KSCN}$ ), or of ammonium ( $\text{NH}_4\text{SCN}$ ), for the determination of small quantities of iron, particularly in commercial aluminium salts, is, of course, common to all laboratories. It has been practised for many years, both for scientific and technical purposes, and, as is well known, depends on the formation of the red ferric thiocyanate ( $\text{Fe}_2\text{6SCN}$ ), soluble in water, alcohol, and ether. It is often recklessly referred to as "the sulphocyanide process," without reference to the marked effect upon the results of alterations in the working conditions, such as amount of reagent used, proportion of free acid present, presence of oxidisers employed to convert ferrous into ferric iron, etc.

In his process the author takes advantage of the solubility of the red colour in ether on the following grounds:—

Ether extraction of the red ferric thiocyanate has the following advantages, under all working conditions: It develops the red tint to triple intensity, besides increasing its brilliancy. It obviates the difficulty too commonly met with, of comparing colours in solutions which are opalescent from mechanical impurities, and in solutions which have naturally a brown or yellow tinge. It admits of the concentration of the iron, by the extraction of a large volume of ferric solution with a small volume of ether, and in this respect it is to micro-iron-determination what distillation is to Nesslerising. Finally, it does away with the injurious effect which aluminium salts have of preventing the development of the red colour, and thus admits of iron being estimated in alum with precision, by comparison with standard ferric solution in water, without the necessity of employing an absolutely iron-free alum for comparison.

On these considerations the author has worked out (and illustrated by a large number of test analyses) the following process, which for simplicity, certainty, and rapidity is unsurpassed, if equalled, by anything in quantitative analysis; while for accuracy it is enough to say that it is capable of estimating iron in alum with the greatest ease to a unit of the fourth place of decimals ( $= .0001$ ) per cent., or one part per million, and, with a little more trouble, to a tenth or even a hundredth of that amount.

1. Make a standard solution of iron-alum ( $\text{Fe}_2\text{3SO}_4, \text{K}_2\text{SO}_4 + 24\text{H}_2\text{O}$ ) by dissolving 8.606 grms. and making up with distilled water to one litre. As the alum contained 11.62 per cent of Fe, this will give a solution containing exactly 1 gm. of metal per 1000 cc. This solution should contain no free acid, and it will keep clear for weeks. Weaker standards become turbid, hence they must be made from this when required.

2. Place 1 gram. of the finely-powdered sample in an ordinary stoppered sample-tube of about 30 cc. capacity, and having three file marks—at  $7\frac{1}{2}$ , 10, and 20 cc. respectively. Add 1 cc. of normal iron-free sulphuric acid, and make up to the lowest mark with pure water. Dissolve the alum by shaking the tube for a few seconds in a beaker of hot water, and when dissolved cool the solution again by holding the tube in cold water for a few seconds more. Add now 0.2 gram. of iron-free ammonium thiocyanate, and make up to 10 cc. with pure water.

3. Place 1 cc. of the standard iron solution of 1 in 100 cc. flask, and make up to the latter volume with distilled water. Now place 1 cc. of this reduced iron standard in a similar stoppered sample tube to that of 2, add 1 cc. of the normal acid, make up to the lowest mark with water, add 0.2 gram. of the thiocyanate, and make up to the 10 cc. with water.

4. Finally, fill up both tubes to the 20 cc. mark with ether and agitate them thoroughly. As soon as the contents settle compare the tints, and, if unequal, of course one or more further trials are made with greater or less quantities of standard iron-solutions as may be necessary. Water takes up about  $\frac{1}{10}$ th of its volume of ether, and it so happens that this retained ether holds its proportion of the ferric thiocyanate; but as this is equally the case with the sample and with the standard, it is of little consequence; but in all delicate experiments the ether solution should be drawn off into graduated cylinders after adding a washing with an equal volume of ether, and compared there in any way most convenient.

*Precautions.*—The necessity for iron-free reagents has already been referred to. The thiocyanate may be prepared pure enough for all ordinary purposes by twice recrystallising the best obtainable in a very feebly acid solution, and washing with ether till the washings are colourless. Although the product is not absolutely iron-free, it appears so under working conditions. The acid may be obtained pure enough by distilling into water at a temperature much below its boiling point, of the purest obtainable oil of vitriol. Of course in all cases the reagents will be tested collectively by a blank experiment. The same precautions must be observed with regard to the vessels employed; all must be thoroughly digested with strong hydrochloric acid, and thoroughly washed before use; they should be kept under a glass shade, and employed for nothing else.

The operations should be conducted with as little light as possible, and certainly never in direct sunlight or even in strong diffused daylight.

Oxidisers should be avoided, particularly peroxide of hydrogen, which, even in small quantity, destroys the red colour. Of course they would be required for ferrous iron, even small amounts, if the ether extraction were not adopted, but not otherwise, as when the ether is employed the iron is completely oxidised to the ferric state, probably by the ozonisation of the air with which the fluid is agitated. So completely is this the case that it is of no moment whether the standard iron solution be made from a ferrous or a ferric salt, provided it contains the same proportion of metal.

#### NOTICE TO OUR READERS.

*Erratum.*—In Professor v. Asbóth's paper on the "Estimation of Starch" in last month's ANALYST, on the last line of page 139, the equivalent of starch to baryta is printed as 4.32, while it should evidently be 4.23, or more strictly 4.2353, because  $C_{12}H_{10}O_5$ . BaO would require  $\frac{222}{105} = 4.2353$ .



# THE ANALYST.

SEPTEMBER, 1887.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual country meeting took place at Leamington on Friday, the 22nd July, Mr. Allen, the President, occupying the chair.

The names of several gentlemen were read a first time for election as members and associates.

The following paper, "On the Examination of Wort and Starch," by Mr. E. W. T. Jones, F.I.C., was read and discussed.

The members present dined together on Friday evening, and on Saturday they drove to Warwick Castle and Stratford-on-Avon, when, owing to the magnificent weather, an exceedingly enjoyable day was spent. The headquarters of the Society were at the Manor House Hotel.

### (1) EXAMINATION OF WORT. (2) ESTIMATION OF STARCH.

By E. W. T. JONES, F.I.C.

*(Read at the Meeting at Leamington in July.)*

LOOKING over my notes, it has occurred to me that a short paper, compiled from those having reference to the above subject, may interest public analysts. Although the work does not come under our official duties, doubtless many like myself have been consulted by private clients on the subject, with a view of rebutting excise charges as to the alleged improper use of sugar.

This subject has more or less occupied my attention for many years, and the more one works at it, the greater do the complications appear, and, with all due and very great respect to the Somerset House chemists, whose work, I am sure, is always conscientious, I have strongly felt, with increasing conviction, that the testimony on which they base their charge of added sugar to wort is of the most inconclusive character. They rely upon what is called

(1st) The specific rotatory power of the solids of the wort, *i.e.*, the deviation in angular degrees  $[\alpha]_D$  or  $[\alpha]_D$  of 10 grms. of the solids in 100 c.c. through 1,000 m.m.

(2nd) The cupric reducing power of the solids of the wort, *i.e.*, the initial reduction with "Fehling" of the wort calculated in p. c. of dextrose on the solids.

These terms will be better elucidated for those unacquainted with the subject by the methods, the use of which I propose to describe in detail.

When a sample of sweet wort is submitted for examination, if salicylic acid has not been added, it is desirable to add some at once for its preservation. 1 grain per fluid oz. I find will keep wort in a quiescent state for months. This gives an additional gravity of 0.7, which correction can be made.

Take carefully the specific gravity at 15.5°C., and divide all above 1,000 by 3.86; for giving grms. of solid matter per 100 c.c. (3.95 is, perhaps, the more correct division, but 3.86 is generally used, and is retained here).

Now take 50 c.c., most carefully measured, of the wort, put into a correct 100 c.c. flask, add 2 c.c. of solution of basic acetate of lead, mix and make the bulk, with distilled water, exactly 100 c.c., pour on to a dry filter and collect the clarified wort, which will be fit to examine in the polariscope.

I use a Soleil-Venkyke-Scheebler, but, of course, it is not essential to use this particular instrument, though personally I like it. Whatever instrument be used the result must be expressed in angular degrees through 1,000 m.m., for 10 grms. of the solid matters of the wort per 100 c.c. This is the specific rotatory power, either  $[\alpha]_j$  or  $[\alpha]_D$  according as the sensitive tint or the yellow sodium light is employed. My results are with the former. Example—a sample of wort was found to have a sp. gr. at  $15.5^\circ\text{C}$ .  $1.077.24$ .  $\frac{77.24}{3.86} = 20.01$  grms. of solid matter per 100 c.c.

*Sp. ry. pr.*—50 c.c. of the wort clarified with the lead solution as described, and diluted to 100 c.c. (hence remember only half the original strength), gave in the polariscope, with the 100 m.m. tube, a scale deviation of  $+30.1$  (three observations agreeing). This scale reading is now multiplied by 0.385 to obtain real angular degrees, and by 10 in order to obtain the rotation for 1,000 m.m. thickness of the solution. Thus  $30.1 \times 0.385 \times 10 = 115.885$ ; the solution, however, contains 10.005 grms. of solid p. 100 c.c. Consequently we have  $10.005 : 10 :: 115.885 : x = 115.8$ , the sp. ry. pr. required.

*Cupric Reducing Power.*—20 c.c. of the clarified wort, being diluted to 100 c.c., 10 c.c. (equal to 1 c.c. original wort) with 35 c.c. Fehling solution diluted with 70 c.c. water and heated in a beaker surrounded by boiling water for 15 minutes, gave after ignition of the precipitate (1) 0.2215. (2) 0.2220 gm. CuO, mean 0.2217; this  $\times 0.4536$  gives a reduction equivalent to 0.10056 gm. dextrose for the solid matter in 1 c.c. of the original wort, viz., 0.2001 gm., calculated into a percentage on this equals 50.25, and this is the cupric reducing power of the wort, proposed, I believe, by O'Sullivan to be shortly designated K.

I would here emphasize what Mr. O'Sullivan has long ago pointed out, that the best way (if not the only correct one) of taking the cupric reducing power of a solution such as wort, is that described by him in the J.C.S., 1876, vol. ii., p. 130, viz., to dilute a suitable amount of the Fehling solution with twice its bulk of water, and after it has attained the proper temperature by standing in boiling water, to add the sugar solution, and digest 14 to 15 minutes in the beaker, surrounded by the water kept boiling. The volumetric method, with its variable time and other conditions, is not admissible in the presence of dextrin or with such solutions as wort.

The Somerset House chemists assert that a genuine malt wort should have a sp. ry. pr. *not less* than 120  $[\alpha]_j$   $_{3.86}$  and a cupric reducing power *not over* 50 or 51.

In the example given the wort was made by myself, under carefully observed conditions, with some of the same malt as used by the brewer who was charged with using sugar. Note the result of the examination of this wort:—

Sp. ry. pr.  $115.8 [\alpha]_j$   $_{3.86}$   
K  $_{5.786}$  50.25.



Some of the defendant's wort, forwarded from Somerset House by his desire, gave on analysis:—

Sp. ry. pr.	116.5
K	50.8

The Somerset House chemists' results, as given in Court, being:—

Sp. ry. pr.	120.0
K	57.2

They made no remark on the sp. ry. pr.; but on the cupric reducing power of 57.2 they based their charge of 1.60 p. c. of added sugar (glucose) to the wort. Now, as all of my experiments were made in duplicate, I assert that the analysis of the wort made by myself, and known to be free from added sugar, absolutely disallows any proof based on this result, of any having been used in the brewing complained of, and the mashing temperature and other conditions in the experimental brewing were those not unreasonable but even likely for the actual working. The malt is somewhat exceptional in giving so low an optical activity, but on looking at the table and the influence on the optical activity by keeping worts it was probably slightly higher when first examined, as the Somerset House result goes to show, but I am convinced that their 57.2 for cupric reducing power is an error caused by using the Fehling solution volumetrically in the presence of dextrin.

I append a table showing

- (1) Difference in character of worts from two different samples of malt.
- (2) Influence of mashing temperature on the wort.
- (3) Influence of time (4 months) on wort preserved by salicylic acid.

I believe that the Somerset House chemists place some reliance for detecting the presence of added cane-sugar to wort on the extra reduction of Fehling after heating in boiling water bath for 15 minutes with 2 p. c. of normal sulphuric acid but all the worts I have examined give a little extra CuO after this process, which is probably due only to the hydrolysis of some of the dextrin, and not to cane-sugar at all. For instance: the K of 50.25 in the wort I made myself, became 57.34, and the sample received from Somerset House went to 56.25 after this treatment.

*Dextrin.*—To estimate this in a wort I take 10 c.c. of the clarified wort, add 16 c.c. normal sulph. acid, dilute to about 70 c.c., and boil on the hot plate for 4 hours in a flask covered by a glass marble, keeping at the same bulk by additions of water; when cool I add 16 c.c. N. soda and make to 100 c.c.

10 c.c. (equal to 0.5 c.c. orig. wort) submitted to the Fehling test, and the CuO calculated for 100 c.c., deduct from this the CuO found in the initial Fehling test, after multiplying it by 1.68436, for the amount of CuO yielded when maltose is converted into dextrose, and multiply the remainder by 0.40816 for dextrin.

Table showing difference in Wort from two samples of Malt, the effect of temperature and time in mashing on the constitution of the Wort, etc.:—

	Worts made by myself in Laboratory.				Publican's Wort alleged to contain Sugar.			Somerset House Analysis of the foregoing Wort.
	From Malt obtained haphazard in Wolverhampton.		From Malt out of same bulk as that used for wort complained of.		Sample forwarded from Somerset House. Analyzed by me.			
Mashed Temperature	2½ hours 135-140° F.	2½ hours 160-165° F.	2 hours 135-140° F.	3 hours 135-140° F.	4 Sep. '85	1 Jan. '86	4 Jan. '86	June, '85
Sp. Gr. at 15.5° C.	1067.49	1070.98	1077.24	1062.24	1079.60	1080.00	1080.00	1080.10
Solids, per 100 c.c. by divisor 3.86	17.48	18.39	20.01	16.12	20.62	20.72	20.72	—
Sp. Rt. Pr. [α]	121.14	134.42	115.8	113.6	116.5	114.4	114.09	120.0
Cupric Reducing Pr.	47.99	39.50	50.25	51.84	50.80	53.01	54.17	57.2
Grammes per 100 c.c.								
Maltose, (i.e., all the initial cupric reduction calculated as such)	...	...	16.08	13.37	—	17.57	...	...
Dextrin	...	...	1.57	1.27	—	1.51	...	...
Albuminous matter, etc. (by difference)	...	...	2.36	1.48	—	1.61	...	...
Percentage on the Solid Matter.								
Maltose, as above computed	...	...	80.36	82.94	—	84.80	...	...
Dextrin	...	...	7.85	7.88	—	7.29	...	...
Other Bodies (by difference)	...	...	11.79	9.18	—	7.91	...	...

*Experiments with Starch.*—As allied to the subject of my wort paper, I propose to give an analysis of a sample of maranta starch (arrowroot) by O'Sullivan's method, and the subsequent treatment of the wort (I may call it) by acid to complete hydrolysis, also the result of treating a portion of the starch directly with acid to complete hydrolysis.

For O'Sullivan's paper see J.C.S., 1884.

Six grammes of arrowroot, being gelatinised in about 80 c.c. water, after cooling to 62° C., 0.04 grm. diastase dissolved in about 2 c.c. of water was added, and the temperature kept 62° to 63° C. for about an hour; the solution was then boiled, and filtered through a weighed filter, the residue left on the filter after washing and drying and allowing for the ash equalled 0.18 per cent. on the arrowroot. The solution being more than 100 c.c. was evaporated, and made exactly 100 c.c. at 15.5° C.; its sp. gr. was 1020.34.

This solution examined by the polariscope through the 200 m.m. tube, gave a deviation of +46.2 scale degrees.

3.021 grms. of the solution gave with Fehling 0.1645 grm. CuO; this equals 5.556 grms. CuO for the 100 c.c.

$5.556 \times 0.7256 = 4.031$  grms. of maltose in the 100 c.c., and as 1 grm. of maltose p. 100 c.c. gives with the 200 m.m. tube a deviation of 8.02 scale degrees, with this instrument 4.031 grms. would give 32.33 scale degrees of deviation as due to maltose; the remainder of the 46.2 total deviation, viz., 13.87, must be due to dextrin, 1 grm. of which p. 100 c.c. with the 200 m.m. tube gives a deviation of 11.56 scale degrees, hence the 13.87 equals 1.200 grms. dextrin. Now we have in the 100 c.c.—



$$\begin{array}{l} \text{Maltose, } 4.031 \times 0.9478 = 3.821 \text{ grms. starch.} \\ \text{Dextrin, } 1.200 \quad \quad = 1.200 \quad \quad \quad \end{array}$$

5.021 grms. of

starch in the 100 c.c., or, from the 6 grms. taken, a percentage of 83.69.

The sample of arrowroot used lost 15.40 per cent. by drying in water oven, and it gave 0.30 per cent. of ash, hence we get—

Moisture	..	..	..	..	15.40
Starch	..	..	..	..	83.69
Fibre	..	..	..	..	0.18
Ash	..	..	..	..	0.30
Loss, etc.	..	..	..	..	0.43
					100.00

Having ascertained that we have in this solution of starch products—

Maltose, 4.031 grms. p. 100 c.c.
Dextrin, 1.200   "       "
Diastase, 0.040   "       "

let us treat it with acid to complete hydrolysis, and see how the chemical method of estimating the dextrin agrees with the optical. About 20 c.c. (weighing 20.4245 grms.) of the solution, being diluted to about 60 c.c., is submitted to hydrolysis by boiling for 4 hours with 16 c.c. normal  $\text{SO}_3$ , then 16 c.c. normal  $\text{NaHO}$  added, and made exactly 100 c.c. — 10 c.c. of this solution yielded with Fehling solution 0.2500 gm.  $\text{CuO}$ , or 2.50 grms. for the 100 c.c., which corresponded to 20.4245 grms. of original solution of the starch

Weight of 100 c.c. of  
the starch products  
found from sp. gr.

products—20.4245 : 102.034 :: 2.50 :  $x$  = 12.49 grms.  $\text{CuO}$  for 100 c.c. of the starch products.

Now from the initial cupric reduction, and that after full hydrolysis, we should be able to calculate the dextrin, and so compare it with the amount found with the polariscope.

The initial cupric reduction of the starch products solution was 5.556 grms.  $\text{CuO}$  for the 100 c.c.—remembering that when maltose is converted into dextrose by hydrolysis the original reduction of 471.276  $\text{CuO}$  for the molecule becomes 793.80  $\text{CuO}$  for the molecule of dextrose formed—we know that the initial reduction of  $\text{CuO}$  due to maltose must be multiplied by 1.68436 to give the amount of  $\text{CuO}$  that would be yielded when converted into dextrose, hence  $5.556 \times 1.68436 = 9.36$   $\text{CuO}$  as due to the hydrolysis of the maltose; this deducted from the total  $\text{CuO}$  found after full hydrolysis, viz., 12.49, leaves 3.13  $\text{CuO}$  as due to the hydrolysis of the dextrin. This multiplied by 0.40816, the factor to convert  $\text{CuO}$  produced from the hydrolysis of the dextrin into dextrose, gives 1.278. So we find—

By the optical and chemical  
method combined.

Maltose, 4.03  
Dextrin, 1.20

By chemical method alone, i.e., initial  
 $\text{CuO}$  and after hydrolysis.

Maltose, 4.03.  
Dextrin, 1.28.

To complete these experiments I treated 1 gm. of this starch mixed with 45 c.c.

H<sub>2</sub>O with 5 c.c. strong HCl and boiled for three hours—neutralised with soda and made exactly 100 c.c. With Fehling this gave 2.056 CuO for the 100 c.c.

$2.056 \times 0.40816 = 0.8392$  grm. starch, or 83.92 per cent., against 83.69 per cent. found by O'Sullivan's method.

As a summary to these statements, I would say that, although genuine malt worts made under ordinary conditions from most malts would have a sp. ry. pr.  $[\alpha]_D^{20}$  of over 120 and a cupric reducing power of under 50, there are malts that give worts having a lower sp. ry. pr. and a higher cupric reducing power under conditions that small publicans may reasonably subscribe to in their brewing, and thus it is unsafe to base a charge of fraud on these data.

My notes on the starch estimations illustrate the exact procedure by O'Sullivan's method, and the full hydrolysis by acid of the solution of starch products of known composition. The mode of calculating the results and their comparison with the former method may prove interesting to many members, as may also the direct full hydrolysis of some of the same starch by acid.

#### UNRELIABILITY OF ANALYSES OF SAMPLES OF MILK WHICH HAVE BECOME PARTIALLY DECOMPOSED BY KEEPING.

C. ESTCOURT, F.I.C., F.C.S.

This matter is becoming of such importance to public analysts generally, that it appears advisable that any special experience on the part of members of our Society should be brought prominently before all interested in the matter.

With this view, I will very briefly give the results of analyses performed at different periods, by three different analysts, upon the same sample of milk.

The first analysis was made by myself as Public Analyst upon the milk purchased 26th February last, delivered to me and analysed the same day. I certified to 14 per cent. added water.

The second analysis was made by a local chemist and druggist upon the portion left with him by defendant on the 12th March.

The chemist who performed the second analysis stated that the milk was quite decomposed and yet stated that he performed three separate analyses, all agreeing. How could he possibly mix a decomposed milk so completely as to be able to take out three separate portions which would give exactly similar percentage results for fat and solids not fat?

This gentleman, whose whole experience in milk analysis had he said extended over three years, had only had a very recent experience of the method of Somerset House, which he used in the present case.

It appears, therefore, more than likely that he did not get off all the fat, and as he did not weigh the solids not fat, the discrepancy is easily explained.

This analyst said no water had been added, but admitted that the sample was adulterated as it had been skimmed.

The third analysis was made by the chemists of the Somerset House Laboratory upon the third portion sent up by the magistrates on the 25th March, at request of defendant.

When the milk reached Somerset House it was undoubtedly in a very decomposed state, and hence this case exhibits the unreliability of the analyses in a very pronounced manner. The Somerset House analysts as usual say, that "after making the allowance for loss by decomposition," they are of opinion the milk has been watered to the extent of 40 per cent.



They do not state whether such allowance is included in the figures.

1	2	3
Analysis of Milk when fresh, 26 Feb., 1887.	Analysis of Milk when decomposed, 12 March, 1887.	Analysis of Milk quite decomposed, 25 March, 1887.
Not fat 7.70	8.77	Not fat 4.79
Fat 3.10	Allowance .40	Fat 1.47
Total solids 10.80	Not fat 9.17	Total solids 6.26
	Fat 2.06	
	Total solids 11.23	

These figures require no further comment.

#### NOTES OF LEGAL PROCEEDINGS INTERESTING TO ANALYSTS.

*(These notes are collected and edited by the Secretaries of the Society of Public Analysts, in virtue of a resolution to that effect, and their publication will be continued from time to time in the portion of the ANALYST devoted to the Society's affairs)*

In a case heard at Lancaster Police-court, a grocer named Wilson was charged with having sold pepper containing 15 per cent. of olive stones, starchy matter, and other foreign vegetable matter. The case brought into Court representatives of the grinders and the wholesale dealers, and these admitted the adulteration, and that the retailer was quite innocent of this adulteration. The defendant had obtained his pepper from a Liverpool firm, who had in turn been supplied with it by Messrs. White, Palmer, and Co., Limited.

Mr. Thomas Vacher Low, the managing director for this firm, said they supplied the pepper and warranted it. He was willing to admit that the pepper complained of was a part of what they supplied, and that the analyst's certificate was more or less accurate. Owing to a fire at a large grinder's in London, they had had a most extraordinary demand for pepper, and were compelled to buy to meet the demand, a thing they had never done before. They got it through brokers, and it was guaranteed as genuine. They took every precaution to have it tested, and sent some samples to Somerset House, where it was declared to be pure, but they had since found that the pepper was adulterated.

At Swansea, there have been some interesting prosecutions for selling beer adulterated with common salt. In one instance, the Borough Analyst, Dr. W. Morgan, certified to the presence of chlorides in a proportion corresponding to 70 grains of common salt per gallon. The sample was referred to Somerset House, whence a certificate was returned affirming the presence of chlorine equal to 67 grains of common salt. Mr. R. Bannister was examined, and stated that some natural waters contained chlorine in greater proportion than this, so that there was no proof of adulteration. No attempt was apparently made to determine the actual sodium chloride present.

In a case heard at Birkenhead, in which Mr. J. Carter Bell condemned a beer as adulterated with water, and evidence was given that there was no standard, and that, therefore, water could not be detected in beer. Mr. Bell states that he found on analysis the original gravity of the beer to be 1043, and consequently wrote to the Inspector, who went to the brewer who supplied the beer. The brewer said he supplied the beer, and when it left the brewery the original gravity was 1051. Hence he had distinct evidence of adulteration, and gave a certificate accordingly that there was 6 gallons of added water in every 36 gallons of beer.

*Conclusion of the Society's Proceedings.*

## ON BECHI'S TEST FOR COTTON-SEED OIL IN OLIVE OIL.

*Abridged from the Report of the Commission of Florence, appointed to examine "Bechi's Test." See "L'Orosi," Feb., 1887, p. 37.*

TRANSLATED BY JOSEPH W. ENGLAND, PH.G.\*

IN a lengthy and exhaustive communication, the Commission of Florence has made public the results of the experiments upon the value of the "Bechi's Test," as a reliable and positive indicator of the presence of cotton-seed oil, fraudulently contained in olive oil. This Commission, appointed in the early part of last year, was as follows: U. Peruzzi, N. Ridolfi, and Prof. G. Roster.

The method of Professor Bechi, as used by the Commission, and with successive modifications by the author, consists in the subjection of a sample of the suspected oil to the heat of boiling water, after first having added an alcoholic solution of silver nitrate, and amylic alcohol and oil of rape, in the manner and proportions hereinafter indicated.

Take one grain of crystallised silver nitrate, and dissolve in the smallest possible quantity of water (about 1 c.c.), and add 200 c.c. of alcohol (96°). The addition, also, of 20 c.c. of sulphuric ether is a good one, in that it makes the reagent better miscible with the oil to be examined, but it is not necessary. On the other hand, prepare a solution composed of 85 parts of amylic alcohol and 15 parts of oil of rape seed. These reagents should be made as needed, and not kept on hand for any length of time.

Now, to apply the test, Prof. Bechi takes 10 c.c. of the oil to be examined, adds 1 c.c. of the alcoholic solution of silver nitrate and then from 8 to 10 c.c. of the mixture of amylic alcohol and oil of rape; agitating strongly, and then heating on a water-bath for 5 or 10 minutes.

In the case of pure oils the colour remains the same as it was after the addition of the reagents. In the event that sophistication has been practised with cotton-seed oil, there will be produced a brownish colour or turbidity, of a varying grade, from a very light brown to a deep maroon or black, according to the quantity of cotton oil present.

With these data furnished by Prof. Bechi, and after having assisted in experiments made by him in support of his method, the Commission instituted a series of long and diligent personal experiments, numbering over 200, in the chemical, biological, and hygienic laboratory of the Royal Institute; adhering strictly to the rules as laid down, measuring exactly, in each instance, the quantities of oils and reagents, and using tubes of equal diameters, or, in one word, employing the same conditions in all experiments, in order to render the result truly comparative.

The oils used were furnished in part by Prof. Bechi and in part by this Commission—looking, especially, for those olive oils of whose genuine nature there could be no possible doubt, and then, secondly, taking good olive oil containing cotton oil. Several of the olive oils were from other countries (Spain, France, Tunis, Dalmatia, and Malta, etc.), but the greater number were from various parts of Italy. Some were recent and some old; others pure of 1a, 2a, 3a quality; and others were rancid.

In order to ascertain if the reaction outlined by Bechi was peculiar to cotton-seed oil addition, alone, the Commission found it necessary to extend their experiments upon other oils, vegetable and animal, alone and admixed with pure olive oil.

\* American Journal of Pharmacy.



Every experiment made was in doubles or triples, that is two samples of the oil (marked No. 1 and No. 3), and another sample of the oil (marked No. 2), which had added to it cotton-seed oil in a certain proportion; subjecting No. 1 and No. 2 samples to the heat of boiling water, after the addition of the reagents, and leaving No. 3 sample without exposure to heat, in order to compare the colours of Nos. 1 and 2 with that of No. 3. The experiments were then especially directed toward the mixture of olive oil with oil of cotton seed. \*The proportion used for the mixture was, generally, 20 per cent. of the latter oil. The Commission held that, if the method of Prof. Bechi will determine any such falsification, it is more than sufficient for any exigency, inasmuch as the fraud practised is always in much larger proportion. The experiments were grouped under five series.

*Series A.*—Cotton-seed oils of various origins.

Eleven samples of oils from the following markets were used:—1. London; 2. New Orleans; 3. Augusta; 4. Louisville; 5. Sample (8 years' old); 6. English (Hirsch); 7. Thorn; 8. Maginnis; 9. Planter's; 10. Aldigè; 11. Creole. All these oils, treated with the "Bechi test," gave a most intensely brownish colour, that exhibited no appreciable variation in shade, according to the origin of the product. The experiments were then repeated upon the oils, using 2 c.c. of oil of cotton to 8 c.c. of olive oil. The olive oil used was from Pons of Scandicci, upon whose genuineness there can be no question.

*Series B.*—Pure olive oil, alone, and admixed with oil of cotton.

The series of experiments here outlined were based upon the first, second, and, in some instances, the third, quality of 48 oils, giving also the origin of each oil. The samples were subjected to comparative tests, alone, and then admixed with 20 per cent. of Hirsch's English cotton-seed oil, the most abundant in Italian markets; in every instance the 48 olive oils, alone, were negatively affected by the reagents, but the instant cotton oil was admixed, and the test then applied, the result was promptly given by the formation of the deep, brownish colours, in every instance.

*Series C.*—Various oils, alone, or mixed with Hirsch's English cotton-seed oil.

The oils here examined are 25 fixed oils of different qualities, liable to be used as an adulterant. They are, for example, oils of rape (Germany, Milan, Marseilles, etc.), sesame (Levant, Georgia, Bombay, Paris, and Grasse), peanuts, poppy, linseed, cocoanut, castor, almond, peach-seed, and cod-liver; and the results show that, in every case, there was no appreciable change with the reagents, but if, prior to the application to the test, 20 per cent. cotton oil was added, the characteristic colour of the reduction was formed.

*Series D.*—Pure olive oil with other fixed oils, alone, and with cotton oil.

These experiments were made to ascertain if the presence of other fixed oils than cotton seed, in a mixture, would have any modifying influence upon the reaction, so characteristic with oil of cotton. It is sufficient to say that the results show that they have none, and the Commission find that the test is therefore limited to that fixed oil alone. The cotton-seed oil was added in 20 per cent. proportion, when used with both oils of olive and benne (in the secondary tests), which latter two were evenly divided (i.e., 40 per cent.), while the oil of sesame was added in equal parts to olive oil, prior to the application of the primary tests.

*Series E.*—Rape oil of different origins, alone, and mixed with cotton oil.

The Commission, in view of the importance that oil of rape obtains, in the application of Bechi's test, examined seven oils of various qualities, derived from different provinces. From these experiments the assertion is made that, while several of the finest samples in the pure, undiluted state, furnished a noticeable change in the formation of a reddish-brown colour, this was always made very much darker if 20 per cent. of cotton-seed oil was previously added; and, on the other hand, if the rape oil examined was previously diluted with pure olive oil or amylic alcohol, as, for example, in the proportions used by Prof. Bechi in his test, *no* change whatever was evinced.

Finally, the Commission, wishing to see if a variation of the proportion of the reagents would more clearly demonstrate results, used a stronger solution of silver nitrate, and found that the brownish colour could be made to vary from brown to black, according to the quantity of the silver salt added. After numerous experiments they decided that the original proportions were the best ones to adopt, in that the test would be much more delicate, and would not, under any circumstances, be caused by the rape oil.

To examine olive oil for admixed cotton oil, with Bechi's method, the Commission recommend the division of the suspected sample into three parts, as follows:—

No. 1. Tube of the suspected oil and reagents.

No. 2. Tube of the suspected oil and 20 per cent. of cotton oil, and the reagents.

No. 3. Tube of the suspected oil and reagents.

Now expose tubes No. 1 and No. 2 to the heat of boiling water for 5 or 10 minutes, but do not heat tube No. 3; use it simply as a guide to see if No. 1 remains unaffected by heat or becomes coloured. If the sample is pure, the oil will remain unchanged, that is, the same in appearance as No. 3, while No. 2 acquires the characteristic colour. If the oil in tube No. 1 has been sophisticated with cotton oil the brownish colouration will soon appear, while tube No. 2 will be a much deeper brown; evidently showing that the brownish colour is due, in part, to the quantity of cotton-seed oil present, as well as the proportion of silver nitrate and oil of rape.

From all that has been presented, and more especially from the clear, concurring, and uniform results obtained in the experiments herein detailed, it is evident that the method proposed by Professor Bechi, used with care, and in the manner indicated, has not failed the Commission in a single instance, and they feel that they cannot do less than to most strongly urge its general adoption, as a reliable and positive indicator for the existence of cotton-seed oil in olive oil, fraudulently added.

In the original article no theory is advanced concerning the chemical reaction that takes place in the application of the "Bechi test," but it seems highly probable that the change is due to a reduction of the silver nitrate to the state of oxide, through the presence of the peculiar yellow colouring principle present in cotton-seed oil. The product, after the testing is finished, measures 11.5 c.c., showing that the residue is simply a mixture of the suspected oil (10 c.c.) and oil of rape (1.5 c.c.), while the alcohols have been totally dissipated by the heat of the water-bath. The utility of the rape-seed oil in the decomposition is not explained, and whether the amylic alcohol, through any chemical change, exerts any influence is also an unsolved problem.



## THE COMPARATIVE DELICACY OF SOME QUALITATIVE TESTS.

By J. S. C. WELLS.

I HAVE so often been shown tests by students, and asked if they indicated any appreciable quantity of the substance tested for, that I have thought it might be of interest to know just how delicate some of the more important tests are.

I have begun with the metals of the fifth group (according to Fresenius), and the accompanying tables show the results obtained. Many tests might, no doubt, have been carried even farther than shown in table by using larger quantities of liquid and allowing them to stand for a longer time. My idea, however, was to obtain results, such as any student might, with the ordinary apparatus used by him. For this reason the tests were all made in the ordinary six-inch test tube, and, unless otherwise stated, were not let stand more than five minutes.

It will be seen from the table that nearly all show, even in very dilute solutions, and that some of them, such as the precipitation of lead by  $H_2S$ , are wonderfully delicate. It should be remembered that the results given were obtained in solutions containing nothing but the substance tested for and the reagent; no foreign substances being present. The results in column I. show the point beyond which it was impossible to distinguish distinct particles of the precipitate. After passing this point the reaction was indicated by a mere cloudiness or colour. In column II. is shown the extreme limit of the test, that is, the most dilute solution in which any reaction was obtainable. In order to get a clear idea of how dilute such solutions are, it may be well to state that one part in one million is equivalent to one grain in seventeen gallons.

## SILVER (Ag).

Reagent.	I.		II.	
	Part of substance.	Parts of water.	Part of substance.	Parts of water.
$NH_4Cl$ .. ..	1	20,000	1	250,000
$HCl$ .. ..	1	20,000	1	250,000
$KBr$ .. ..	1	20,000	1	200,000
$KI$ .. ..	1	5,000*	1	200,000
$H_2S$ .. ..	1	5,000	1	800,000
$K_2Cr_2O_7$ .. ..	1	3,000	1	3,000

## MERCURY (Hg).

$Hg_2O$ .				
$NH_4Cl$ .. ..	1	25,000	1	200,000
$HCl$ .. ..	1	25,000	1	200,000
$H_2S$ .. ..	1	5,000	1	1,000,000
$NH_4OH$ .. ..	1	25,000	1	25,000
$SnCl_2$ .. ..	1	50,000	1	200,000
$K_2Cr_2O_7$ .. ..	1	20,000	1	100,000

$HgO$ .				
$H_2S$ .. ..	1	2,000	1	1,000,000
$NH_4OH$ .. ..	1	4,000	1	4,000
$SnCl_2$ .. ..	1	50,000	1	200,000
$Cu$ .. ..	1	10,000†		

\* Only shows on standing some minutes.

† Shows in solution of 1-100,000 after standing four hours.

LEAD (Pb)				
Reagent.	Part of substance.	I. Parts of water.	Parts of substance.	II. Parts of water.
HCl .. ..	1	500	1	500
NH <sub>4</sub> Cl .. ..	1	600	1	600
H <sub>2</sub> S .. ..	1	20,000	1	1,000,000*
H <sub>2</sub> S in KOH solution ..	—	—	1	1,000,000*
H <sub>2</sub> SO <sub>4</sub> .. ..	1	10,000	1	20,000†
K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> .. ..	1	10,000	1	100,000
BISMUTH (Bi).				
H <sub>2</sub> S .. ..	1	13,000	1	1,000,000
NH <sub>4</sub> OH .. ..	1	10,000	1	12,000†
H <sub>2</sub> O ‡ .. ..	1	10,000	1	20,000
K <sub>2</sub> SnO <sub>2</sub> § .. ..	1	40,000	1	20,000
COPPER (Cu).				
H <sub>2</sub> S .. ..	1	2,000	1	1,000,000
NH <sub>4</sub> OH .. ..	—	—	1	80,000
K <sub>4</sub> FeCy <sub>6</sub> .. ..	1	2,000	1	1,000,000
CADMIUM (Cd).				
H <sub>2</sub> S .. ..	1	4,000	1	200,000
H <sub>2</sub> S + HCl    .. ..	1	20,000	1	200,000
K <sub>4</sub> FeCy <sub>6</sub> .. ..	—	—	1	4,000

Qualitative Laboratory, School of Mines.

### THE POLARIZATION OF MILK.

By H. W. WILEY, CHEMIST TO THE U.S. DEPARTMENT OF AGRICULTURE.

#### I.—SPECIFIC ROTATORY POWER OF MILK-SUGAR.

CRYSTALLIZED milk-sugar when first dissolved possesses a higher rotatory power than it has in the milk from which it was derived. This increased optical activity may be compared with the original by the ratio 8:5, nearly. After the solution has stood for twelve to twenty hours, or immediately on boiling it, this extra rotatory power is lost. In estimating the specific rotatory power of milk-sugar the numbers given always refer to the constant, and not the transient, gyrotory property.

Among the earliest numbers assigned to the rotation of lactose are those of Pogiale ( $a_D = 54.2$  and Erdmann ( $a_D = 51.5$  [Sucrose ( $a_D = 66.5$ ]. Biot ¶ places this number for lactose at 60.23, and Berthelot \*\* at 59.3 for the transition tint ( $a_D$ ). Hoppe-Seyler, in his "Handbuch der physiologisch-chemischen Analyse," gives this number at ( $a_D$ ) = 58.2. Since the ratio of ( $a_D$ ) to ( $a_D$ ) is 1:1.1306, the above numbers become for

\* Seen only on looking down the tube.

† Only on standing a few minutes.

‡ This test was made on a solution of Bi(NO<sub>3</sub>)<sub>3</sub> in presence of NH<sub>4</sub>Cl.

§ This is the test with KOH and SnCl<sub>2</sub>, yielding Bi<sub>2</sub>O<sub>3</sub>.

|| I found that the addition of a few drops of HCl made the precipitate separate much better than it would in either alkaline or neutral solutions.

¶ Compt. Rend., vol. 42, p. 349.

\*\* Würtz Dict. de Chim., vol. 2, 1st part, p. 188.



Biot  $(a)_D = 53.27$ , for Berthelot  $(a)_D = 52.47$ , and Hoppe-Seyler  $(a)_D = 51.48$ . Hesse\* observed the rotation number to be  $(a)_D = 52.67$  when the solution contained 12 grams. per 100 c.c. and the temperature was  $15^\circ \text{C}$ . On the other hand, when the concentration is only 2 grams. per 100 c.c. the number assigned is  $(a)_D = 53.63$ . It appears from this that the specific rotation power of a solution of milk-sugar diminishes with the increase of its concentration, and this view is adopted by Landolt, Tollens, and Schmidt.

The following general formulat† is used to correct the reading of the polariscope for concentration of solution:—

$$(a)_D = 54.54 - .5575c + .05475c^2 - .001774c^3,$$

in which  $c$  = number grams. sugar in 100 c.c. solution. These observations are contradicted by the work of Schmoeger,‡ who, in an elaborate series of experiments, using instruments of different construction and observing all necessary precautions, found the rotation number of lactose sensibly constant for all degrees of concentration up to the saturation point. In thirty-two series of investigations, in which the degree of concentration gradually increases from  $c = 2.3554$  to  $c = 36.0776$ , and in which a constant temperature of  $20^\circ \text{C}$ . was maintained, the variations in the numbers obtained were always within the limits of error of observation. The mean of all these numbers fixes the value of  $(a)_D$  at 52.53.

According to Schmoeger variations in temperature have far more to do with changes in rotatory power than differences of concentration. The value of  $(a)_D$  falls as the temperature rises. Under  $20^\circ \text{C}$ . the disturbing influence of temperature is greater than above  $20^\circ \text{C}$ . At the latter degree  $(a)_D$  varies inversely about .075 for each  $1^\circ \text{C}$ . change of temperature. Pellet and Biard,§ as a result of their observations, fix the rotatory power of milk-sugar at 58.94 for  $(a)_D$  [ $\delta$ ,  $(a)_D = 52.12$ ].

After a careful review of the methods used in the above *résumé* and the numbers determined by them, I am inclined to accept the mean obtained by Schmoeger as the one entitled to the greatest credit. It also has the advantage of being almost the mean of all the various numbers which have been assigned as the specific rotating power of lactose, viz. :—

Poggiale	..	..	..	..	..	..	..	..	54.20
Erdmann	..	..	..	..	..	..	..	..	51.50
Biot	..	..	..	..	..	..	..	..	53.27
Berthelot	..	..	..	..	..	..	..	..	52.47
Hoppe-Seyler	..	..	..	..	..	..	..	..	51.48
Hesse	..	..	..	..	..	..	..	..	52.67
Hesse	..	..	..	..	..	..	..	..	53.63
Schmoeger	..	..	..	..	..	..	..	..	52.53
Pellet and Biard	..	..	..	..	..	..	..	..	52.12
Mean	..	..	..	..	..	..	..	..	52.65

In the present state of our knowledge, therefore, the specific rotatory power of milk-sugar should be taken at  $(a)_D = 52.5$ . I propose, at an early date, to make a careful study of this subject, in order to fix, if possible, an exact number for the expres-

\* Anal. Chem. u. Pharm., vol. 176, p. 98.

† Tucker, Sugar Analysis, p. 91.

‡ Ber. chem. Gesell., vol. 12, p. 1222 *et. seq.*

§ Bull. de l'Assoc. des Chimistes vol. 1, p. 171 *et. seq.*

sion of the rotating power, and to examine the conflicting evidence respecting the influence of the degree of concentration on the same. The estimation of lactose in milk by the polariscope is rendered difficult also by the presence in milk of various albumens—all of which turn the plane of polarization to the left. As will be seen by the data given further along, the ordinary method of removing these albumens, viz., by a solution of basic lead acetate, is far from being perfect. If, therefore, a portion of the albumen be left in the liquid submitted to polarization, the rotation to the right will be diminished by its presence.

Hoppe-Seyler\* assigns as the rotation power of egg albumen  $(a)_D = -35.5$ , and for serum albumen  $(a)_D = -56$ . Both acids and alkalies seem to increase the rotating power, which may with acetic acid reach  $(a)_D = -71$ .

Fredericq† gives the rotation number for blood serum for the rabbit, cow, and horse at  $(a)_D = -57.3$ , and for the dog at  $-44$ . Paraglobulin, according to the same author, has a rotation number  $(a)_D = -47.8$ .

Milk albumen‡ has the following numbers assigned to it:—

Dissolved in $MgSO_4$ sol.	$(a)_D = -80$
Dissolved in dil. HCl.	$(a)_D = -87$
Dissolved in dil. NaOH sol.	$(a)_D = -76$
Dissolved in strong KOH sol.	$(a)_D = -91$

The hydrates of albumen§ have rotation powers which vary from  $(a)_D = -71.40$  to  $(a)_D = -79.05$ . From the chaotic state of knowledge concerning the specific rotating power of the various albumens, it is impossible to assign any number which will bear the test of criticism. For the purposes of this report, however, this number may be fixed at  $(a)_D = -70$  for the albumens which remain in solution in the liquids polarized for milk-sugar.

The phenomenon of "birotation" in milk-sugar has already been noticed. The problem of analysis of this sugar is, however, still further complicated by the facts pointed out by Schmoeger|| and Erdmann,¶ that when milk is rapidly evaporated in a plain dish the sugar is left in the anhydrous state, and that this sugar in fresh solutions exhibits the phenomenon of "half rotation." When such sugar is extracted with alcohol and re-evaporated, it, doubtless, is still anhydrous. But in the calculation of results this sugar is generally estimated as containing water of crystallization, and thus an error, which Schmoeger reckons at as much as .2 per cent., is introduced into the results. This fact, not well recognised, combined with the knowledge that in the process of evaporation many particles of sugar must be occluded by the hardening caseine, tends to throw doubt upon the accuracy of estimating the sugar by the extraction method.

The work which I undertook had for its object the determination of the best method of preparing the milk-sugar solution for the polariscope, and a comparison of the numbers obtained by this instrument with those given by the ordinary process of extraction.

\* Würtz, Dict. de Chimie, vol. 1, 1st part, p. 91.

† Compt. Rend., vol. 93, p. 465.

‡ Hoppe-Seyler in Handbook of the Polariscope, Landolt, p. 248.

§ Kühne and Chittenden, Am. Chem. Jour. vol. 6, p. 45.

|| Ber. chem. Gesell., vol. 12, 1915 *et. seq.*; vol. 13, p. 212 *et. seq.*

¶ Ber. chem. Gesell., vol. 12, p. 2180 *et. seq.*



The reagents used for removing the albumens were:—

- (1) Saturated solution basic lead acetate, specific gravity 1.97.
- (2) Nitric acid solution of mercuric nitrate diluted with an equal volume of water.
- (3) Acetic acid, specific gravity 1.040, containing 29 per cent.  $\text{HC}_2\text{H}_3\text{O}_2$ .
- (4) Nitric acid, specific gravity 1.197, containing 30 per cent.  $\text{HNO}_3$ .
- (5) Sulphuric acid, specific gravity 1.255, containing 31 per cent.  $\text{H}_2\text{SO}_4$ .
- (6) Saturated solution sodium chloride.
- (7) Saturated solution magnesium sulphate.
- (8) Solution of mercuric iodide in acetic acid; formula\*  $\text{KI}$ , 33.2 grams.  $\text{Hg Cl}_2$ , 13.5 grams. Strong  $\text{HC}_2\text{H}_3\text{O}_2$ , 20.0 c.c. Water, 64.0 c.c.

Alcohol, ether, and many solutions of mineral salts, hydrochloric, and other acids were also tried as precipitants for albumen, but none of them presented any advantages which would make a detailed account of the experiments of any interest.

Table No. 9 contains a record of the experiments which led to the adoption of 1 c.c. acetate of lead solution, or 1 c.c. acid mercuric nitrate, as the best amount of each for 50 c.c. of milk.

Nearly all the polarisations were made in a 400 m.m. tube. From two to four observations were made with each sample. An average of these readings was taken for each determination. In the calculations the value of  $(a)_D$  was taken at 53 instead of 52.5, the number which subsequent investigations have led me to believe more exact. The instrument employed was a "Laurent Large Model" polariscope.

In all cases the volume of the solution was corrected for the volume of the precipitated caseine. The volume was assumed to occupy 2 c.c. for each 50 c.c. milk.

Since in the Laurent instrument the weight of sucrose in 100 c.c. to read even degrees on the scale is 16.19 grams. [ $(a)_D = 66.67$ ], it follows that the weight of lactose in 100 c.c. to read one degree on the scale for each per cent. lactose present would be  $16.19 : x = 53 : 66.67$ ;  $x = 20.37$ .

If 52.5 be taken as the value of  $(a)_D$  for lactose, then  $x = 20.56$ .

In table No. 9, A indicates acetic acid, Pb basic acetate of lead, MR acid mercuric nitrate, etc. The letters C and H indicate the temperature—C denoting the ordinary temperature of the room, and H that the sample was heated to  $100^\circ \text{C}$ . and cooled before filtering.

The numbers obtained by extraction with alcohol are taken as the basis of comparison, not because I believe them to be more reliable, but because that method is the one generally employed in the estimation of milk-sugar.

In the alcohol extraction the milk was evaporated to dryness in a thin glass capsule, the dish and dried residue pulverised in a mortar, washed with ether into a continuous extraction apparatus, exhausted with ether, and then with 80 per cent. alcohol for ten hours.

Duplicate analyses are indicated in the table by the small brackets.

\* Jour. de Pharm. et de Chim., vol. 10, p. 108.

TABLE NO. 9.—Percentage of milk-sugar.

Number.	Per cent. lactose extracted by alcohol.	Reagents employed in precipitating albumens.						
		Pb 1cc.	Pb 2cc.	Pb 3cc.	Pb 4cc.	Pb. 5cc.	A 5cc.	Other reagents.
		Per cent. lactose.						
1	4.57				(10°.)	3.67	4.23	
2	4.52				2.45	3.57	4.14	
3	4.46	4.48					3.57	
4	3.92	4.19				3.35		
5	4.35		3.55				4.32	
6	3.71	4.01					3.00	
7	4.10	{ 4.63 4.96 H					{ 4.44 4.68 H	
8	4.16	{ 4.29 4.33					{ 3.80 3.67 H	
9	4.48	{ 4.59 4.58 H					{ 4.04 4.12 H	
10	4.10	4.12 H					3.47 H	
11	4.80	4.87 H			(4°.)		4.44 H	
12	4.77	5.02 H	4.82 H	4.50 H	4.31 H			H <sub>2</sub> SO <sub>4</sub> 4°. 4.70 H 5°. 4.76 H 6°. 4.76 H 8°. 4.76 H
13	4.25	4.25	3.75	3.38	3.38		3.97	3°. 3.89 HNO <sub>3</sub>
14	4.22	4.90 H	4.58 H					
15	3.14	{ 4.40 4.32 H					4.68 H	4.66 H 4.60 H
16	3.30	4.43 H						
17	4.72	4.45	4.18	3.87	3.65	3.26	3.96	3.98 { 3.98 H 4.27 4.43 H <sub>2</sub> SO <sub>4</sub> 4.59 H
18	4.88	{ 4.87 4.87 H					{ 4.37 4.25	(5°.) 4.51 4.43 H { 4.79 H 4.59 H
19	4.31	{ 4.71 4.86 H					{ 4.43 4.43 H	
20	4.39	4.11						
21	4.70	4.17						
22	4.96	{ 4.93 4.97 H					{ 4.45 4.43 H	4.69 4.67 H
23	4.60	{ 4.41 4.45					{ 3.86 3.86	3.90 3.94 3.90 4.10
24	4.74	{ 4.41 4.45 H					{ 4.21 4.35 H	4.32 4.32 4.44 H 4.55 H
25	4.59	{ 4.33 4.37 H	{ NaCl 1°. MR 4.29 H	4.29 H			{ 3.94 3.93 H	{ 4.03 4.10 H
26	4.39							3.98
27	4.60	4.18 H						
28	4.26	3.67 H						

*Remarks on Table No. 9.*—The results obtained by using various other reagents for precipitation of the caseine, viz., MgSO<sub>4</sub>, CuSO<sub>4</sub>, HCl, etc., have not been entered in table. In none of these cases was there sufficient encouragement to warrant an extension. In most cases the precipitation was slow or imperfect, and the filtration difficult.

One important fact should not be overlooked, viz., that any excess of basic plum acetate causes a rapid decrease in the rotatory power of the solution; whether the decrease is due to precipitation of the sugar or solution of the albumens does not clearly appear. Illustrations of this decrease are seen in analyses 2, 12, 13, and 17.

*It seems to make little difference whether the precipitation is made hot or cold.*



The question of temperature is set forth in greater detail in the next table. From all the experiments made it clearly appeared that the best optical results are obtained by the use of a minimum quantity of basic lead acetate, or of either the acid mercuric nitrate or iodide. For 50 c.c. to 60 c.c. of milk, 1 c.c. of the lead acetate or mercuric nitrate solution of the strength noted, and 25 c.c. of the mercuric iodide solution are the proper quantities. It makes no difference, however, if a large excess of the two latter reagents is employed. Of the three the last is to be preferred.

In Table No. 10 will be found the results of the comparative determinations of milk-sugar by extraction with alcohol, by precipitation with 1 c.c. basic lead acetate, and the same with 1 c.c. acid mercuric nitrate, hot and cold, to each 60 c.c. of milk.

In many of the analyses the large differences in results by the three methods show a fault of manipulation, but all the results have been given without selection.

TABLE NO. 10.—Percentage of milk-sugar.

No.	Reagents employed.					No.	Reagents employed.				
	Extracted by alcohol	C.	H.	C.	H.		Extracted by alcohol	C.	H.	C.	H.
		Pb. 1cc.	Pb. 1cc.	MR. 1cc.	MR. 1cc.			Pb. 1cc.	Pb. 1cc.	MR. 1cc.	MR. 1cc.
	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.		Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
1	4.55	....	4.74	....	4.92	34	4.37	4.65	4.93	4.93	....
2	4.10	....	4.22	....	4.50	35	4.52	4.27	4.41	4.56	....
3	4.51	4.54	4.22	4.68	4.62	36	4.88	4.83	4.93	5.17	....
4	4.36	4.55	4.53	4.89	4.90	37	4.61	4.30	4.43	4.57	....
5	4.05	4.14	4.09	4.48	4.39	38	4.79	4.59	4.67	4.91	....
6	3.84	....	3.84	3.98	3.98	39	4.67	4.26	4.41	4.51	....
7	4.52	4.67	4.73	5.01	5.00	40	4.79	4.64	4.74	4.94	....
8	4.25	4.21	4.26	....	4.51	41	3.95	4.10	4.26	4.38	....
9	4.45	4.61	4.54	4.87	4.87	42	4.00	4.61	4.61	4.77	....
10	4.92	5.20	5.22	5.43	5.47	43	4.63	4.24	4.37	4.57	....
11	3.84	....	3.72	4.00	3.96	44	4.77	4.64	4.70	4.94	....
12	4.53	4.61	4.64	4.87	4.85	45	4.85	....	4.53	4.73	....
13	4.57	4.54	4.55	4.91	4.84	46	4.71	....	4.67	4.93	....
14	4.66	4.29	4.45	....	4.63	47	4.34	4.06	4.12	4.40	....
15	4.17	3.65	3.75	3.95	3.87	48	4.05	4.67	4.77	4.83	....
16	5.02	4.66	4.64	4.86	4.86	49	3.67	4.12	4.18	4.36	....
17	4.68	4.03	3.94	4.39	4.37	50	3.78	4.58	4.62	4.82	....
18	4.23	3.82	3.89	4.08	4.02	51	4.19	4.27	4.57	4.53	....
19	4.96	4.70	4.84	4.04	5.04	52	3.83	4.68	4.78	4.97	....
20	4.85	4.39	4.41	4.53	4.65	53	3.86	3.97	4.07	4.21	....
21	4.63	4.47	4.47	4.69	4.67	54	4.59	4.59	4.61	4.83	....
22	4.47	4.39	4.45	4.67	4.71	55	4.02	4.26	4.36	4.40	....
23	4.46	4.23	4.31	4.65	4.63	56	4.36	4.62	4.76	4.94	....
24	4.47	4.59	4.67	5.01	4.95	57	4.20	4.18	4.28	4.48	....
25	4.40	4.41	4.55	4.45	....	58	4.09	4.52	4.56	4.74	....
26	4.85	4.67	4.73	4.97	....	59	4.09	....	4.28	4.46	....
27	4.45	4.21	4.33	4.57	....	60	4.12	....	4.49	4.81	....
	4.44	3.98	4.10	4.28	....	61	4.20	....	4.33	4.41	....
	4.10	4.21	....	4.55	....	62	4.45	....	4.25	4.77	....
	4.38	5.57	4.69	4.89	....	63	4.33	....	4.09	4.37	....
	4.20	4.21	4.37	4.57	....	64	4.62	....	4.33	4.99	....
	4.69	4.59	4.67	4.89	....						
	4.52	4.27	4.41	4.41	....	Av.	4.33	4.34	4.38	4.58	4.63

In the following table will be found the percentage of milk-sugar obtained by using varying quantities of the mercuric iodide reagent, and a comparison of the results obtained with those given by the use of acid mercuric nitrate and basic plumbic acetate:—

TABLE NO. 11.—*Percentage of milk-sugar.*

Number.	Reagents employed.					
	Pb.	MR.	Mercuric iodide.			
			20cc.	25cc.	30cc.	35cc.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
1	4.28	4.48	4.56	4.56	....	....
2	4.46	4.57	4.62	4.66	....	....
3	4.37	4.65	4.63	4.63	4.60	4.65
4	4.37	4.53	4.60	4.53	4.63	4.60
5	4.38	4.63	4.50	4.53	4.53	4.59
6	4.33	4.67	4.43	4.53	4.60	4.66
7	4.30	4.67	4.67	4.67	4.59	4.57
8	4.33	4.59	4.50	4.53	4.50	4.59
9	4.27	4.60	4.63	4.60	4.66	4.66
Av.	4.34	4.60	4.57	4.58	4.61	4.62

## II—ALBUMEN REMAINING IN FILTRATE FROM LEAD ACETATE AND MERCURIC IODIDE SOLUTIONS.

From the fact that the polariscopic readings show that solutions of milk prepared with lead acetate have a lower rotating power than those prepared with mercury salts, it is to be inferred that the lead reagent either leaves certain soluble and transparent kinds of albumen in solution, or else dissolves a portion of those which are at first precipitated. To test the accuracy of this supposition a few analyses were made to determine the amount of albumen left in the filtrate from the lead and mercury reagents. At the same time different quantities of the mercuric iodide solution were used, in order to determine the amount which would give the best results. For 60 c.c. milk the quantity of mercuric iodide to be used should be 25 c.c. to 30 c.c.

In the following table will be found the percentages of albumen in the whey after precipitating with the reagents noted and filtering. Ten cubic centimetres of the filtrate were evaporated to dryness in a thin glass dish, and the dried residue (with the glass) burned with soda lime. The calculated nitrogen was then multiplied by 6.25 and the product taken as the percentage of albumen:—



TABLE No. 12.—*Per cent. albumen in filtrate.*

From Pb.	From HgI <sub>2</sub> . 15cc.	From HgI <sub>2</sub> . 20cc.	From HgI <sub>2</sub> . 25cc.	From HgI <sub>2</sub> . 30cc.	From HgI <sub>2</sub> . 35cc.
·0865	·1950	·0865	·0865	·0562	·0865
·1130	·0674	·0865	·0865	·0865	·0865
·1130	·0674	·0562	·1130	·0562	·0312
·0865	·0674	·0562	·1130	·0562	·0562
·1130	·0674	·0562	·0312	·0865	·0562
·1130	·0090	·0865	·0300	·0865	·1412
·1130	....	·1412	·1130	·1412	·0562
·1950	....	·1412	·1130	·1412	·1412
·1130	....	·1412	·0865	·1412	·0865
·1412	....	·1130	·1412	·1130	·0865
·1130	....	·1362	·0090	....	....
....	....	·1250	....	....	....
....	....	·0090	....	....	....
....	....	·0090	....	....	....
Av. . . ·1182	·0789	·0888	·0839	·0964	·0828

In table No. 13 will be found percentages of albumen remaining in filtrate from acetate precipitation of forty-two samples taken from those represented in table 10. From these two tables it is at once seen that the quantity of lævo-rotatory matter remaining in milk after treatment with basic lead acetate is much greater than those samples treated with the two mercuric salts. This explains at once the higher per cent. of milk-sugar obtained by using the last-named reagents, and shows that the use of lead acetate as a clarifying agent must be abandoned:—

TABLE No. 13.—*Per cent. albumen after precipitation by lead acetate.*

Number.	Per cent.	Number.	Per cent.	Number.	Per cent.
1	·250	16	·237	31	·329
2	·306	17	·237	32	·305
3	·135	18	·169	33	·305
4	·272	19	·103	34	·237
5	·134	20	·271	35	·305
6	·239	21	·237	36	·339
7	·301	22	·271	37	·237
8	·305	23	·235	38	·374
9	·237	24	·271	39	·203
10	·339	25	·237	40	·373
11	·271	26	·237	41	·305
12	·305	27	·271	42	·339
13	·267	28	·339	Av. . . .	·278
14	·237	29	·350		
15	·271		·374		

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

To the Editor of the ANALYST.

SIR,—I observe that in a recent contribution to the *Farmatzevtichesky Journal* (No. 12, 1887, p. 177), Professor Loesch, of St. Petersburg, has described a process for the estimation of theine in tea, consisting in the extraction of the tea-leaves with boiling water, evaporation of the infusion on a waterbath after addition of magnesia, and extraction of the residue with chloroform.

This process, as many other chemists who, besides myself, must have used it can no doubt testify, is an excellent one, but it is certainly not new. I have employed it for the past six years (with the modification of extracting the tea-leaves in a flask provided with a vertical Liebig's condenser), and like it better than any other process for the determination of theine. It answers, of course, equally well in the analysis of coffee.—Faithfully yours,

E. GODWIN CLAYTON, F.C.S., F.I.C.

## OLEOMARGARINE AND MICE.

To the Editor of the ANALYST.

DEAR SIR,—It may interest the readers of the ANALYST to know that mice seem to possess the power of distinguishing between oleomargarine and butter.

The facts are these: Some time ago I received for analysis a lot of butters whose genuineness was questioned. The dishes in which the butters, preparatory to analysis, were melted, were, after the fat had been roughly poured off, permitted to remain over night on one of my working tables. The next morning I was surprised to find that ten of the twelve dishes had been licked clean by mice, while two had been left untouched. My analyses finished, it turned out that all the suspected samples were pure butter with the exception of two, and, upon inspection, I discovered that these two were the very fats the mice refused to eat. It seemed hardly possible that this could be due to mere luck, so I spread out in a clock glass a lot of butter, and in another and similar glass a lot of oleomargarine. The glasses were placed along side each other on my table and allowed to remain over night. Next day I found the glass which had contained the butter polished as clean as though it had been well washed, whereas the one charged with oleomargarine was to all intents and purposes intact. The mice had merely nibbled at it. I next set out four glasses, two containing butter and two oleomargarine. The result in this case was also conclusive. The mice dined on the two butters and completely ignored the oleos. The oleo in one glass was untouched, and that in the other merely bore the footprints of a mouse. When no butter, but simply a glass of oleo was left on the table, the mice partook thereof, 'tis true, but only in small quantities. There is no doubt that a hungry mouse will eat oleo, but it seems that he will do so only on condition that there is no butter to be had. The fact is a curious one and shows what a keen capacity for discrimination a mouse possesses. I could not possibly have told previous to my analyses which fats were oleomargarine and which butter. How would it do to make this extraordinary acuteness on the part of mice the basis of a rough method for testing butter? Methinks the words "mouse test" would look and sound remarkably well. The mice could be kept in a cage, and the suspected fats temptingly displayed on watch-glasses arranged on a little shelf running from one end of the cage to the other.

Perhaps some mice are incapable of making the distinction, and will eat oleo as greedily as pure butter. I have had no experience with mice other than with those which hang about my laboratory, consequently I am unable to say what would happen should a trial be instituted with a lot of outsiders.

Here is a chance for original research! Let those who have time, opportunity, and the requisite modicum of enthusiasm fall to. Perhaps there may be a future for the mouse test!—Yours,  
L. W. MCCAY.

John C. Green School of Science, Princeton, N.J., U.S.A.

## APPOINTMENT.

At the Annual General Session of the Peace for the county of Lancashire, held at Preston on June 30th, Mr. Walter Collingwood Williams, of Liverpool, was appointed deputy analyst of food and drugs for the county, at a salary of £150 per annum, and a fee of 8s. for every certificate of analysis.

Dr. J. Campbell Brown is still the head analyst for the county, at a salary of £300 per annum and fees.



# THE ANALYST.

OCTOBER, 1887.

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### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

#### THE VISIT OF THE SOCIETY OF PUBLIC ANALYSTS TO THE FARMS OF THE AYLESBURY DAIRY COMPANY ON THE 9TH JUNE, 1887.

IN the July issue of the ANALYST it was briefly mentioned that a number of members of the Society, in virtue of an invitation from the Directors of the Aylesbury Dairy Company, spent the day, subsequent to the June meeting, at the said Company's farms, near Horsham, Sussex, and I have thought that we still owe to our absent members a fuller account of this very interesting visit.

To commence with a few general notes, the rise and progress of the Aylesbury Dairy Company may not be without interest.

When in the year 1865 the cattle plague invaded this country, and also visited the cow-sheds of London, necessitating the slaughter of a great number of milch cows, Mr. G. Mander Allender, who for some time had paid great attention to practical dairy-farming, formed the idea of supplying London with really good milk, direct from the country. In order to carry this scheme into effect, he, in 1865, founded this Company, of which, since that time, he always has been, and still is, the most active and energetic managing-director. The business was increasing so rapidly that it was soon found to outgrow its first abode, and in 1870 it was removed to the specially-built and extensive premises at St. Petersburg Place, Bayswater.

From the very commencement of the business, the supply of pure milk of high quality was made the leading principle. To completely secure the purity of the milk, it was necessary to have a certain control over it from the time it leaves the cow—nay, even more, over the conditions under which the cows are kept, and by which they are surrounded. The Aylesbury Dairy Company, therefore, only enters into a contract for the supply of milk, after the farm in question has been inspected and satisfactorily reported upon by the Company's sanitary engineer, as well as by the local medical

officer of health, with regard to sanitary conditions of cow-sheds, milk rooms, etc., special attention being paid to drainage and purity from contamination of the water used for cleansing purposes. Further regulations in the contract, which the farmer desirous to supply the Company with milk is obliged to sign, provides for the utmost cleanliness in handling the milk, the notification of the occurrence of contagious diseases in the district, on the farm, or in the house of any person employed on the farm. If, in consequence of any disease existing on the farm, it should be deemed advisable to stop the supply of milk, the sender is guaranteed to sustain no loss whatsoever. On the other hand, the farmer is liable to heavy fines in the case of neglecting the provisions of the contract.

It is scarcely necessary to say that at the Bayswater premises the most perfect arrangements exist for most thoroughly cleansing, by means of boiling water, steam, etc., of everything coming in contact with milk. The whole of the employes and the premises are under the supervision of a medical officer, to whose knowledge every case of sickness, no matter however slight, is brought. Every man in the employ of the Company is also bound to inform the secretary of any case of infectious disease occurring in his family or the house he lives in.

Every churn of milk entering the dairy has always been tested by thermometer and lactometer for temperature and specific gravity, and, previously to 1880, a number of samples were sent regularly once a week to an eminent analyst. But this system did not seem a sufficient protection, and in the year mentioned a fully-equipped analytical laboratory was established, and put under the charge of Dr. Vieth. Here a more extended and thorough control over the milk passing through the business is executed than exists anywhere in the world. Attendants at the Society's meetings and readers of the ANALYST are well acquainted with the nature and extent of the work accomplished in this laboratory through Dr. Vieth's annual reports.

It is obvious that for various reasons it must be considered a drawback to such a large concern to be entirely dependent on purchased milk. The desirability of being not distributors only, but also producers of milk, made itself felt more and more by the Aylesbury Dairy Company, and in 1884 led to the purchase of the Itching field Estate, comprising an area of 1,400 acres, and divided into six holdings, which, after they had successively come in hand, were all thrown into one well-arranged farm. The farm is situated in the county of Sussex, within  $1\frac{1}{2}$  miles to the south of the town of Horsham, and is intersected by the Mid-Sussex line of the London, Brighton, and South Coast Railway. The general character of the land is that of a readily workable loam, resting on a calcareous sandstone, which secures a good natural drainage. The estate has the advantage of a constant supply of pure water. This is obtained from a lake, fed from springs which have never been known to fail in any season. From this lake the water is pumped up to a tower, and also into a large reservoir, holding half-a-million gallons; thence the buildings and fields are supplied by gravitation. In no instance are animals allowed to drink from ponds. The buildings are, with very few exceptions, new constructions of a suitable and commodious, though by no means extravagant, character.

To this estate, then, the visit of the 9th of June was paid. The visitors were met by



Col. Talbot and Mr. Burls, Directors of the Aylesbury Dairy Company, at Victoria Station, where a special train was in waiting, taking the company to Horsham, and thence to the private siding on the estate. The numerous company included the President, Vice-Presidents, Secretaries, and a good many members of the Society of Public Analysts, as well as many Medical Officers of Health, and a number of other gentlemen, among them Mr. Lennox Peel, C.B. ; Mr. Stephen Terry, C.E., Local Government Board ; Professor Brown, C.B. ; Mr. Cope, Agricultural Department, Privy Council ; Professor Robertson, Principal, Royal Veterinary College ; and others.

On quitting the train the visitors were received by Mr. Allender, the Managing Director and founder of the Company, and on their way to the chief farm (Stammerham) had an opportunity of inspecting a number of animals representing the various breeds kept on the estate, which were paraded, their respective characteristics being pointed out. There were pure shorthorns, and what are known as "dairy" shorthorns, animals not entitled to be entered in the herd book, and yet many of them highly bred. Shorthorns are perhaps the most useful all-round animals for the dairy farmer, producing as they do, if properly selected and fed, milk good both in quantity and in quality, and are yet useful as beef-makers. Then came polled Norfolks, hornless, as their name implies—grand red animals. Mr. Allender thinks highly of this breed, and is inclined to increase the number of them on the Company's farms. Next, the pretty Jerseys, which, if not yielding as much milk as the larger shorthorns, far surpass all other breeds for quality, seven per cent. of fat being by no means uncommon in the milk of these little animals. A still smaller cow, the Kerry and Dexter Kerry, are almost a speciality on the Company's farms, their herd of these useful little beasts already numbering sixty or seventy. They give an astounding quantity of milk for their size, and it is of excellent quality. Sussex cattle are also kept, as they are the cattle of the district, and although essentially a beef-producing breed, give milk of good quality, although in small quantity. Each breed is kept pure, part of the Company's business being the sale of cattle, as well as the production of milk. The herd at present numbers nearly 400 head, and it was stated that it will be shortly increased to 500.

Pigs, of course, are maintained as consumers of the by-products of a dairy. The Tamworth, a pig of a sandy red colour, is principally kept ; and indeed this breed is a speciality of the Company, its chief point being a large percentage of lean meat, as distinguished from the terribly fat animals—masses of lard—one usually sees. Very great numbers of these pigs are exported by the Company, chiefly to the north of Europe—Germany, Denmark, and Norway. Southdown sheep, and any quantity of poultry of various pure breeds, complete, with exception of the horses, the live stock of this large farm.

After inspecting the cattle a move was made to the Dairy. This is plain, substantial, and very spacious, light and air being secured to the highest degree. The drainage is perfect, simply because there is not a drain in the whole range, everything in the way of liquid finding its exit direct into the open air. The floors are impervious, being specially constructed of a mixture of the best Portland cement and sharp sea grit—a mixture which not only becomes as hard as granite in the course of a short time, but is capable of being made perfectly smooth and thoroughly water-tight.

Here the milk is brought direct from the cow-houses. If required to be sent to London as milk, it is poured into a large vessel holding some 500 gallons, and from thence it passes over the refrigerators by which it is deprived of all animal heat, and thence flows into the cans in which it travels to London. If cream be required, the milk is passed through a centrifugal separator which enables the cream, in a perfectly fresh condition, to be despatched to London within two hours of the time the cows are milked. The skim milk is at once sent off to that portion of the Company's estate where the celebrated herd of Tamworth pigs is kept, and thus the young pigs are rapidly grown into valuable animals.

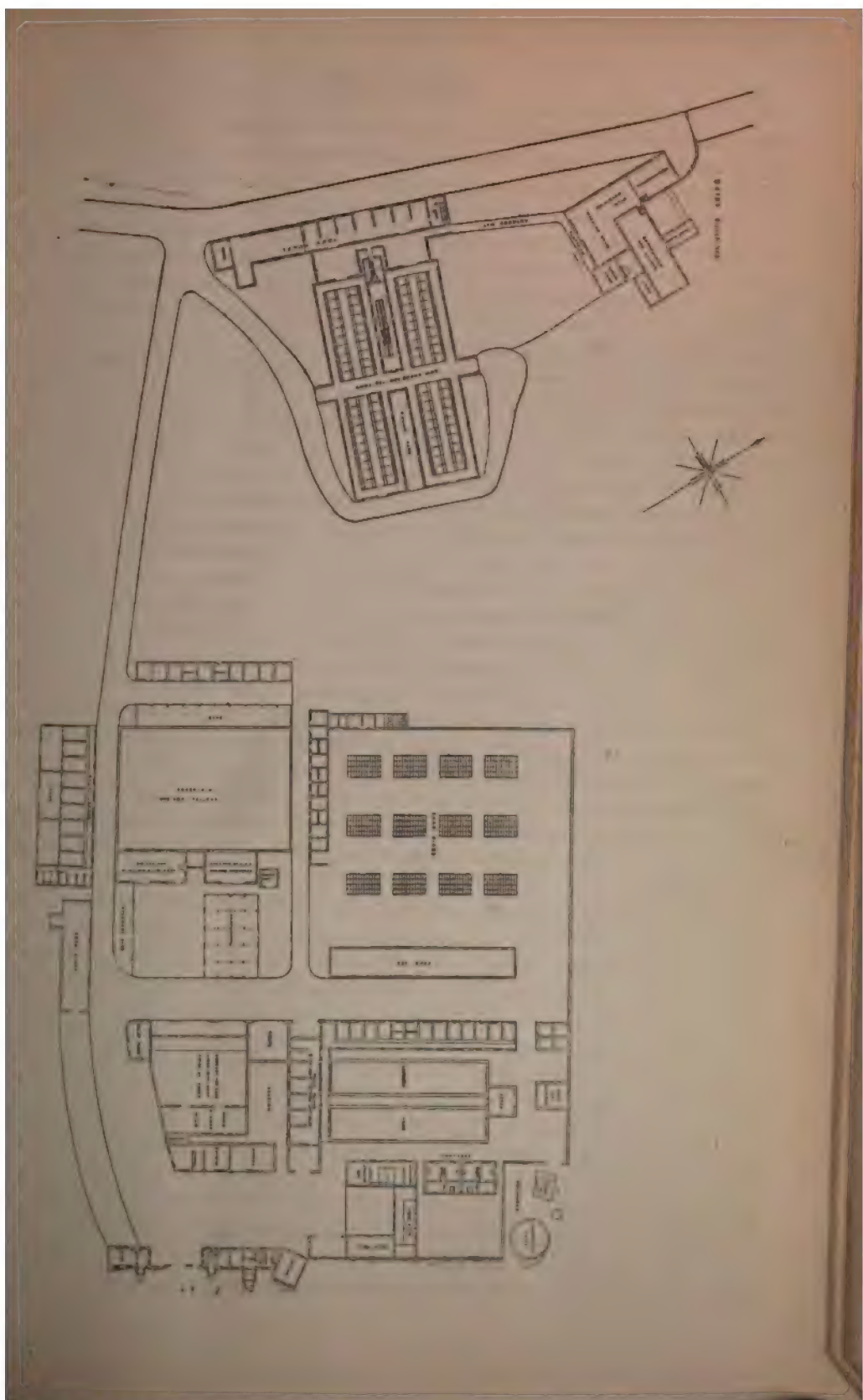
On leaving the dairy the visitors found themselves in the first of the pair of cow-houses, in each of which eighty cows are kept. The houses, designed and built by Mr. Allender (as indeed are all the buildings) are especially worthy of notice. Space, light, and ventilation have been most carefully studied. The dimensions are 172 feet long, 40 feet wide, and 24 feet high to the lantern, which extends the whole length of the houses, thus affording to each of the eighty cows over 1,200 cubic feet of space, or 50 per cent. over and above that fixed by the Metropolitan Board of Works. In addition to ten large windows on each side of the houses the lantern is lighted throughout its entire length. The mangers are of hard Staffordshire ware, and in order to guard against any possible chance of stale food ever being left, between each meal a constant stream of water is allowed to flow through the mangers. With exception of the men and boys actually employed in attendance upon the cows, no other persons are permitted to enter the houses, except in company of some chief official, and none of the ordinary farm hands are ever allowed in. The milkers are inspected every week by the medical officer of the Company, and a certificate is signed by this gentleman as to their sanitary condition. Lavatories and eating-rooms are provided.

There are a great number of other extensive buildings upon the Company's farms (as shown on the accompanying map), notably a covered yard, supposed to be the largest in England, capable of housing one hundred head of cattle; a fine range of bull boxes, containing nine bulls of different pure breeds; stables, etc., etc. The Stammerham buildings alone cover an area of nearly ten acres, and form perhaps the largest homestead in the country.

After the inspection of the buildings the Company were entertained at luncheon, Mr. Allender occupying the chair. Alluding to the paper read by Dr. Klein on the 27th ult. before the Royal Institution, Mr. Allender said that if the cow were the terrible creature represented by Dr. Klein, all who were interested in dairy work, and in cow-keeping especially, must take measures for observing the signs of disease in the animal, and for preventing the dissemination of the evils alluded to by Dr. Klein. The Company were now breeders of their own stock, and with the exception of male animals they had purchased all the cattle they would ever buy. Every sanitary care was taken of their stock, and the great object of the Company was to provide for the public a thoroughly reliable milk supply. He hoped that that would not be the last visit to Horsham of the public analysts and medical officers of health.

The President of the Society of Public Analysts (Mr. ALLEN) said he desired on behalf of his Society (and he hoped he would be supported by their colleagues, the





medical officers of health) to return thanks to Mr. Allender and those associated with him in the Aylesbury Dairy Company, for their very courteous reception that day, and for the very thorough inspection they had been permitted to make of everything on the premises. Speaking as he did for the Public Analysts, he thanked the Company for the opportunity which had been afforded them of thoroughly studying the practical production of milk on the largest and most favourable scale. Many years ago he saw in *Punch* some specimen questions on useful knowledge, and among them was the question: "To what are we indebted for milk?" Answer: "The pump and the chalk pit." He had not seen a genuine old-fashioned pump on the premises, and although he had looked well for the chalk pit he had failed to discover it. The Chairman had alluded to the Public Analysts' standard for milk as being 11·5 per cent. of total solids. That body had never suggested anything like that figure. They had suggested 11·5 as the *lowest limit*, not the *standard*; and it was a great misfortune that people sometimes mixed up the two things. As was known to those present, the Aylesbury Dairy Company's product averaged 13 per cent. of solids, and he believed that amount to be representative of really wholesome milk, such as could be obtained from properly fed and healthy cows. It was the misfortune and not the fault of Public Analysts that, because there were here and there cases of worn out or badly fed cows that yielded only 11·5 per cent. of solids, they were, therefore, legally compelled to pass over many cases where initially rich milk had really been watered to some extent. When he got out of the train he thought he was in Arcadia, and on going through the premises he saw that he was in Hygeia, and he was bound to confess that these two words were not always synonymous as they were in the present case. He would like to be allowed to add a word respecting the Company's chemist, Dr. Vieth. That gentleman was an honoured member and a Vice-President of the Society, and they owed to him some of the most admirable researches on milk extant. He had under his hand the most complete material for the purpose, and both the Analysts and the public were indebted to him for an enormous amount of information he had contributed to the Society's proceedings on the subject.

Dr. WYNTER BLYTH, Medical Officer of Health, St. Marylebone, expressed his satisfaction at the way in which large companies took notice of the researches which were made as to scarlet fever in connection with milk. He had always insisted that if we were to drink milk at all the supply must be managed by large companies. The precautions which were necessary could not be observed in oil shops, grocers shops, and such places, where milk was now so much sold. In his own family the milk used was always boiled, and he believed that the milk of the future would be sterilised milk—that was milk heated to 200 degrees for a few hours.

Dr. CHARLES KELLY, Medical Officer of Health (West Sussex), spoke of the dangers attending milk by being brought into contamination with impure water.

PROFESSOR G. T. BROWN said there was no question at all that milk was a ready carrier of many forms of contamination. The next point was as to the existence of a disease in the cow which should be in the human being transmuted into scarlatina. When Mr. Power's report was first brought under his notice, the Lord President of the day, Earl Spencer, put on the paper a minute directing him (the speaker) to make



an investigation, and since then the investigation had been more or less continuous. At that time, however, the proposal was that a disease in the cow which was not scarlatina—at least, that was what he had understood from the report—would become changed into scarlatina in man. In Dr. Klein's lecture, as he (the speaker) understood it, the proposal was that the cow was subject to scarlatina from the human being, and being so subject, might transmit the disease to man. ("No, no.") Well that was what he had gathered from Dr. Klein's observations, and he might for a moment express his extreme regret that Dr. Klein—he meant the *Times* reporter (laughter)—had not expressed Dr. Klein's view sufficiently clearly to enable him to decide, from a simple reading, what Dr. Klein intended to convey on the matter, which was simply tremendous in importance to the agricultural interest. When the editor of the *Lancet* committed himself to the statement that the experiments which Dr. Klein had made and Mr. Power's report placed the question beyond the region of controversy, he had committed himself to a statement which no bacteriologist of any importance in the civilised world would endorse. The idea of anything connected with bacteria being out of the region of controversy was worthy of the pages of *Punch*. Another journal had put the matter into the region of controversy by a single sentence. The *British Medical Journal* said, wait until we publish the report of Dr. Jamieson. When the question was brought into something like practicable shape, it would then undoubtedly appear that investigations of very prolonged and very intricate character would be necessary. They would have to find cases of this disease, which he believed to be about the most common one that occurred in the cow. He, however, fully agreed with Dr. Klein in his remarks on the importance of rendering the infective matter of various diseases which milk would carry harmless by a process of heating. As to a remark by Dr. Wynter Blyth, the Aylesbury Dairy Company might sterilise their milk and guard it from infection in every possible way, but that would not protect it from contamination by the retail dealers. [Mr. ALLENDER: Our Company are themselves the retailers, and we have complete control over our milk.] There was no more deadly fluid, taken on the average, than milk, except water; and as both of them took a very much higher range than any alcoholic fluid, it became a serious question whether dealers in milk, and, if there were such persons, dealers in water, should be properly licensed. It had been very gratifying to look on what they had seen that day, every conceivable method being in operation for the purpose of preventing the contamination of the milk.

Dr. HENRY ARMSTRONG, Medical Officer of Health, Newcastle-on-Tyne, said he had been amply rewarded by what he had seen for the long journey he had made to be present that day. One of the facts he would be able to take home with him was that the Aylesbury Dairy Company recognised that 1,200 cubic feet of space was desirable for housing a cow. He had been struggling to obtain in his town 800 cubic feet.

Mr. ERNEST HART said he thought that sterilisation would be a very admirable remedy for a great many of the evils of this country. Sterilisation would have prevented the necessity for a great many recent Acts of Parliament. It would have prevented a great deal of crime; and applied to speech, it would have prevented a great amount of waste of time in Parliament and other assemblies. He could not think that sterilisation was a principle of any extensive lacteal application, or necessary to any organisation based on sound principles.

The first principle of any sound undertaking was wholesome production, to avoid the necessity of sterilisation. He had had the honour of being connected with the Aylesbury Dairy Company for a great many years, and he thought he might say for it that its first element, and the basis of its whole organisation, was not sterilisation, but sound production. Some one had said that the two most dangerous drinks were milk and water, but there was nothing that was useful in nature that was not dangerous, and indeed there was no single act of life that was not dangerous if it were abused. The first duty of those having charge of milk or dairies was to see that there was no poison at the source. They had heard a great deal about the filtering of the Thames water, but that was after sewage had been introduced into it. They knew that it was the first duty of Parliament to prevent the introduction of pollution, and the Aylesbury Dairy Company had always acted on the idea that those whose duty it was to produce milk ought to present an article of sound, wholesome, and high quality. They had heard something of the standard of milk, and of the minimum standard. So long as they had a minimum standard, there would always be a commercial tendency to work down to it, but this Company had always considered it to be its duty to work to the highest. All that they had heard that day about microbes, germs, and scarlet fever, was not to the point. He would not say that it was a simple waste of time, as they were matters that had to be studied; but in connection with this Company, the great duty it set before them was to put the cow under such conditions that from the cow (and including the cow) to the consumer, there should be no possibility of the introduction of germs. For that purpose they had to take a healthy cow, to keep it under healthy conditions, and to see that their receptacles and everything coming into contact with the milk were kept free from pollution. This done, they could well afford to leave the rest to the two departments of Government, who, he was sorry to see, instead of combining with each other in consultation, had appointed professors to fight with each other. He hoped that those who had joined in the inspection of the farm this day would see that there was a combination of wealth and correct scientific knowledge, to produce milk which was distributed direct from the cow to the consumer, which required no sterilisation, and was perfectly free from all disease germs. It was the intention of the Company to have no diseased cows, and *that* should be the main object of every dairy company.

Dr. J. F. J. SYKES (Medical Officer of Health, St. Pancras) also addressed the meeting, and a vote of thanks was afterwards passed to Mr. Allender, on the motion of COLONEL the HON. W. P. TALBOT.

After luncheon the company were taken to the calving farm, and to another farm, at which the pigs are kept, in every place much being seen worthy of admiration. This walk, some distance away from the principal homestead, Stammerham, afforded an opportunity of seeing some of the corn and fodder crops and of the pastures; and an ascent of Sharpenhurst Hill revealed a charming panorama of a large part of Sussex. It was then time to take to the train, and a merry homeward journey brought to a close a most interesting, instructive, and enjoyable day.

*Conclusion of the Society's Proceedings.*

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COMMUNICATION FROM THE SOUTH LONDON PUBLIC LABORATORY—  
SIMPLE SUGGESTIONS FOR THE PURCHASE AND ASSAY OF COMMERCIAL CARBOLIC COMPOUNDS USED FOR SANITARY PURPOSES.

By JOHN MUTER, F.R.S.E., F.I.C., AND L. DE KONINGH, F.I.C.

Owing to the extensive use of these compounds by the various local sanitary authorities throughout the country, and the occasional demands on the official analyst for their valuation, it is very desirable, both in the interests of the producers and the consumers, that there should be some simple and definite methods agreed upon for their analysis. Up till the present time we venture to think that if the same sample were sent to any dozen analysts, taken by chance all over Great Britain, not more than two or three would come within measurable distance of each other in the percentage of carbolic acid found, while it is more than probable that the manufacturer's chemist would disagree with all. This state of things is caused:—

(1) By the fact that, very probably, nearly all the operators would employ a process of their own devising, and that, although most would work upon similar lines, yet the details would be so different as to cause a very wide divergence in the finished results.

(2) By the fact that, in commercial mixtures of carbolic acid and the higher phenols, the commonly adopted methods come entirely to grief.

The process of Koppeschaar (although we continually meet with some one who has modified it so as to render it perfect) is totally unreliable for commercial use, because it is full of pit-falls. For example, to begin with, we have the necessity of working on such small quantities that any experimental error is multiplied by something like 1,000; and then, as we are really to a great extent precipitating cresol and calculating it as phenol, we cannot on the face of such a fact expect much truth in the result. This is not, however, all, because the process is not to be depended upon even when dealing with pure medicinal acid. One may set to work to make a series of test experiments, and may theorise that, if it can only be arranged so that we work with the same excess of bromine and for the same time, we are bound to get concordant results. If any one has succeeded in really carrying such reasonable theory into practice without meeting with some unaccountable differences in the very first long series of experiments, then he has been much more lucky than we have ever been, and, so far as we know, we have tried every published modification, besides those resulting from our own experiments.

Another thing which tends to lead to much dissatisfaction in such matters is the tendency of local authorities to contract wildly for powders or liquids "containing (say) 15 per cent. carbolic acid," without getting their medical officer to ask the analyst whether such an article really exists in commerce. The manufacturer takes such a contract with a light heart, because he knows that he can practically put in an acid as tarry and weak as he likes, and argue (if challenged) that he really put in 15 per cent. of "commercial" carbolic acid, and bring reliable witnesses, if needful, to prove they saw it done. Meantime the powder is complained of and sent to the analyst, who is asked to state the percentage of carbolic acid. He does this according to his lights, and naturally takes the words of the contract as literal, and reports the amount of *real phenol* he finds. This being generally microscopic, the authority becomes indignant, and

the manufacturer, in defending himself as above shown, hints that the analyst is incompetent, and generally gets one or two persons on the local board to believe him. If, in such matters, the boards could be induced to consult their analyst before issuing the form of tender, they would then learn that words like those we have above commented upon are absolutely useless in such cases, and that it is ridiculous to ask for a thing which never could be supplied in practice within any reasonable limit of cost.

It may be now taken as a tolerably well admitted fact that, setting aside the necessities of surgery and medicine, the cresol and other higher phenols are just as good for ordinary antiseptic purposes as real carbolic acid itself, and that unless the presence of such congeners be permitted, the cost of disinfectants of this class would be quite prohibitory. It is therefore sufficient that the analyst should see that his board is being supplied with an article which is all made up of mixed phenols, and not with tar oils, containing only a small proportion of such bodies. To ensure this the following, or some similar, form of tender should be employed: "Commercial liquid carbolic acid at — per gallon. This article is not to be darker than pale sherry, and it is to contain not less than 95 per cent. of carbolic and other allied acids obtained from tar, the said acids being in their liquefied form, but free from any excess of water. The said article to be subject to the approval of the Public Analyst (whose decision shall be final) on a sample drawn from each delivery by the Medical Officer. Such analysis is to be carried out and the result communicated to the contractor within six days after delivery of each consignment, and should the result be unfavourable the contractor shall remove the rejected article at his own charge."

Turning now to carbolic powders, we may take it as also a fairly admitted proposition that a commercial acid, such as has been above described, may justly be used in their manufacture, provided that the base of the powder is some siliceous matter, or gypsum, or native limestone, with none of which phenols will combine; but when such acid is employed the use of slaked or caustic lime should be rigorously tabooed. It has been proved that the antiseptic powers of cresol (for example) are entirely masked when combined with an alkali or alkaline earth, and what is true about this body is probably also correct about the other higher phenols present. Analysts looking after the interests of their district should advise a specification worded something like the following:—"Carbolic powder, containing not less than 15 per cent. of the commercial carbolic acid as above described, and having for its base any siliceous or other inert mineral matter with which the acid will not combine. The use of quick or slaked lime as bases are specially to be avoided, and each consignment is to be subject to the same conditions of delivery and approval as in the case of the acid above described."

In some cases a small supply of really pure liquefied carbolic acid is required, and then the simplest contract is for an article which shall correspond in every respect to *acidum carbolicum liquefactum* of the British Pharmacopœia.

Such specifications are now in force in several of the districts served by this laboratory, and since their adoption, together with the processes about to be described, there have been no disputes; moreover, on the few occasions when a parcel had to be rejected, the manufacturer, on making inquiries, found that his own people had been in error, and at once admitted the mistake.



There is nothing more involved in the processes than the following well-known data:—

(1) Phenol, cresol, and their homologues are completely soluble when shaken up with a 5 per cent. solution of sodium hydrate.

(2) Liquefied phenol and the corresponding cresol are insoluble in a saturated solution of sodium chloride.

(3) In the presence of a sufficient excess of alkali, even a largely diluted solution may be boiled down without the slightest appreciable loss of phenol or cresol.

(4) Tar oils and naphthaline are only very slightly dissolved by the alkali, and may be perfectly removed from the solution by agitating it with benzol.

Taking these ascertained facts, we apply them in the following manner, taking each case in turn:—

(1) PROCESS FOR THE VALUATION OF A CARBOLIC POWDER IN WHICH THE BASE IS NOT LIME, AND THE PHENOLS ARE CONSEQUENTLY NOT IN COMBINATION.

Weigh out 100 grammes of the powder, and transfer the same to a flask, and add 400 c.c. of methylated spirit; then, having introduced a well-fitting cork, agitate for a minute or two at intervals during an hour, and finally set the whole aside to settle. When subsidence is complete, pour or filter off 300 c.c. of the supernatant liquid, which thus represents 75 grammes of the original powder actually taken for analysis. Here it may be noted that in laboratories fitted with the specially large "Soxhlet" apparatus required, a more rapid and economical method is to mix the original powder with bran, and to extract it in such an apparatus, with just sufficient spirit to do the necessary work.

To the spirituous extract of the powder, obtained as above described, 200 c.c. of 5 per cent. solution of sodium hydrate are now added, and the mixed liquids are evaporated to about half their bulk. At this point any tar-oils or naphthaline present will separate out, and are to be removed by filtration. The filtrate, freed from these impurities, is now evaporated down to a bulk not exceeding 50 c.c., and transferred to a specially constructed measuring tube surrounded by cold water, the basin being rinsed out with water, so that the entire amount of fluid in the tube shall be exactly 65 c.c. The tube employed is of peculiar form, capable of holding over 110 c.c., and is specially graduated and stoppered. It is wide at the base, and is narrowed at 65 c.c. from the bottom to such a diameter as to show, when graduated, .25 of a c.c. The first graduation is at 20 c.c. from the bottom, the second at 65 c.c., and then the delicate graduations (in .25 of a c.c.) commence and continue for 45 c.c. more up to 110 c.c. The tube is furnished with a long stirring rod made as thin as possible, and projecting above the tube to a convenient extent. [These tubes are to be procured from Mr. Cetti of Brooke Street, Holborn, who knows them as "Muter's carbolimeters," and the entire apparatus costs 8s. 6d.] Before proceeding to use the tube for the first time, it is necessary to find the allowance to be made for the rod. This is done by filling the tube with brine up to the 75 c.c. mark and then pouring in liquefied carbolic acid up to the 85 c.c. mark. The rod is now introduced and the amount of displacement it causes noted, so that an equivalent allowance may be made on all future experiments.

The measuring tube having been charged, as already described, we now proceed to

add very cautiously, and with constant stirring 25 c.c. of strong hydrochloric acid, and when that is all in, we follow it with a teaspoonful of common salt. All the phenols now rise to the surface, and when the whole is at the temperature of 60° Fah., the volume is read off, which gives the amount of commercial carbolic acid present in the 75 grammes of powder. If a perfectly accurate result be required, it is necessary to remove some of the floating acid with a pipette, take its specific gravity, and correct the volume reading to weight.

(2) MODIFICATION OF THE ABOVE PROCESS FOR USE WITH POWDERS ON A LIME BASE, WHERE THE ACIDS ARE COMBINED.

Before proceeding with the spirit extraction, the weighed portion of the powder is to be treated in a capacious mortar with successive small quantities of diluted sulphuric acid (one in three) until the whole mass has a faintly acid reaction, and then the process applied as above described. It is necessary to be very particular about the treatment in the mortar, because, if the least particle of the powder escapes the action of the acid, the results are vitiated, while at the same time, any marked excess of acid should be avoided. Both pestle and spatula must be thoroughly used.

(3) EXAMINATION OF A SAMPLE OF COMMERCIAL CARBOLIC ACID.

*Case 1. The sample is dark in colour.*—Put some of the sample into the "carbolic-meter" up to the 20 c.c. mark, then gently add 5 per cent. solution of sodium hydrate up to the 100 c.c. mark, and lastly add 10 c.c. of benzol. Put in the stopper, and having inverted the tube once or twice, plunge it into cold water. Repeat this shaking and cooling until the separation is complete. Read off the volume of the dark layer, which will now have formed beneath the 100 c.c. mark, and the amount of this will give the tar-oils, etc., present in the sample. Provided there is no excess of water, this amount is deducted from 20 c.c., and the difference multiplied by five gives the percentage strength of the sample. Excess of water (which is of very rare occurrence) is best ascertained by adding some of the sample to three times its volume of benzol, when it should dissolve quite clear if there be no such excess.

*Case 2. The sample is not darker than pale sherry.*—Try if it dissolves nearly clear in four times its volume of 5 per cent. solution of sodium hydrate, and if so, it may be taken as practically free from tar-oils. If not, treat it as above, using however, only a very small fixed volume of the benzol. This acid is apt to contain excess of water, which must be estimated by shaking 20 c.c. of the sample in the "carbolic-meter" with 80 c.c. of saturated solution of sodium chloride, and observing the diminution in volume that will take place if such excess be present.

A fair idea as to whether the acid is chiefly phenol or cresol may always be obtained by applying the bromine reaction, and observing the nature of the precipitate.

(4) EXAMINATION OF A SAMPLE OF PURE CARBOLIC ACID IN THE LIQUEFIED FORM.

In examining samples of this acid, specific gravity practically goes for nothing. The points to determine are:—(1) That it should entirely dissolve to a perfectly clear solution in four times its volume of 5 per cent. solution of sodium hydrate; (2) That, when shaken with an excess of saturated solution of sodium chloride, as already described, it suffers no diminution in volume; (3) When treated with bromine water in excess it gives a fine curdy precipitate, not at all inclined to stick to the tube.



In conclusion, we can only repeat our statement that, if those persons engaged in the examination, and in the general supervision of carbolic antiseptics supplied to public authorities, would only agree to universally adopt some such simple methods and specifications as we have described, there would be universal satisfaction, instead of the disputes now so common, and, in addition to all that, the public money would be spent to infinitely better purpose than it now is in too many districts. We could name at least one district in the Metropolis itself where the present fever epidemic is being fought with so-called "carbolic acid," which is practically all inert tar-oils, and does not contain even 2 or 3 per cent. of the higher phenols, much less any real carbolic acid. It is time that local authorities consulted the public analysts more frequently before entering into such a purely chemical matter as a contract for carbolic articles. It is not to be expected that medical officers can keep such things in order, unless where (as in certain of our own districts) the knowledge of the chemical officer is called into play to check the contractors, and to advise as to forms of contract that can really be carried into practice with full justice to all parties concerned.

#### THE POLARISATION OF MILK.

By H. W. WILEY, CHEMIST TO THE U.S. DEPARTMENT OF AGRICULTURE.

(Concluded from page 181.)

#### III.—COMPARISON OF RESULTS OBTAINED BY EXTRACTION WITH ALCOHOL AND POLARISATION.

By consulting table No. 10 it will be seen that the percentage of sugar obtained by extraction with alcohol is practically the same as that got by polarisation of the lead acetate filtrate.

Thus, the mean percentage of sugar by alcohol (65 analyses) is 4.32; by lead acetate, cold (53 analyses) is 4.34; by lead acetate, hot (64 analyses) is 4.38; by mercuric nitrate, cold (61 analyses) is 4.58; by mercuric nitrate, hot (24 analyses) is 4.63.

If now the milk-sugar, as has already been intimated, exists in an anhydrous state after extraction with alcohol, the percentage of it after the addition of the molecule of water would be increased. Thus molecular weight of anhydrous milk-sugar, 342: molecular weight of the hydrous 360 = 4.38 :  $x$ , whence the value of  $x = 4.61$ . This agrees very nearly with the number obtained by acid mercuric nitrate.

By a study of table No. 13 it is found that the mercuric iodide gives nearly the same rotatory power as mercuric nitrate, and also by combustion the filtrates from the milks clarified by lead acetate contain more albumen than those prepared with mercuric iodide. There is, therefore, every reason for believing that the numbers given by the mercury salts are nearer the truth than those from the lead.

It may be urged that the increased rotatory power observed by the mercury salts is due to the conversion of the dilute acids of a part of the lactose into galactose, which has a rotatory power greater than that of milk-sugar. But when it is remembered that the quantity of acid introduced is extremely minute, that the samples need not be warmed, that they can be filtered and polarised within a few minutes of the time of the introduction of the reagents, the suggestion is seen to be of no force.

For example, in the acid mercuric nitrate it was found that the percentage of sugar was the same whether one, five, or ten cubic centimetres of the reagent were employed and whether it was polarised immediately or after heating and cooling. It is evident that 1 c.c. of the reagent, containing less than a half cubic centimetre of nitric acid diluted in 100 c.c. of liquid, could not exert any notable effect on the rotatory power of the solution.

In the mercuric iodide solution 20 c.c. of acetic acid are used for every 660 c.c. of the reagent.

Thirty cubic centimetres of this reagent contain, therefore, about 1 c.c. of acetic acid. This, in 100 c.c. of liquid, immediately filtered and polarised, could not affect in any marked degree the rotatory power.

Since combustion with soda-lime shows that the filtrate from the mercuric iodide sample is practically free from albumen, it is evident that the numbers obtained in this way must be a near approximation to the truth.

#### IV.—THE PROCESS OF ANALYSIS.

The reagents, apparatus, and manipulation necessary to give the most reliable results in milk-sugar estimation are as follows:—

*Reagents.*—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. One cubic centimetre of this will precipitate the albumens in 50 c.c. to 60 c.c. of milk.

(2) *Acid mercuric nitrate*; dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. One cubic centimetre of this reagent is sufficient for the quantity of milk mentioned above. Large quantities can be used without affecting the results of polarisation.

(3) *Mercuric iodide with acetic acid* (composition already given).

*Apparatus.*—(1) Pipettes marked at 59.5 c.c., 60 c.c., and 60.5 c.c. (2) Sugar flasks, marked at 102.4 c.c. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

#### V.—MANIPULATION.

(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be any given degree. The work can be carried out equally well at 15° C., 20° C., or 25° C. The slight variations in rotatory power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of milk is determined. For general work this is done with a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flask.

(3) If the specific gravity be 1.026, or nearly so, measure out 60.5 c.c. into a sugar flask. Add 1 c.c. of mercuric nitrate solution, or 30 c.c. mercuric iodide solution, and fill to 102.4 c.c. mark. The precipitated albumen occupies a volume of about 2 c.c. Hence, the milk solution is really 100 c.c. If the specific gravity is 1.030, use 60 c.c. of milk. If the specific gravity is 1.034, use 59.5 c.c. of milk.

(4) Fill up to mark in 102.4 c.c. flask, shake well, filter, and polarise.



## VI.—NOTES.

In the above method of analysis the specific rotatory power of milk-sugar is taken at 52.5, and the weight of it in 100 c.c. solution to read 100 degrees in the cane-sugar scale at 20.56 grammes. This is for instruments requiring 16.19 grammes sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk-sugar whatever instrument is employed.

Since the quality of milk taken is three times 20.56 grammes, the polariscopic readings divided by 3 give at once the percentage of milk-sugar when a 200 m.m. tube is used.

If a 400 m.m. tube is employed, divide reading by 6; if a 500 m.m. tube is used, divide by 7.5.

Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method gross errors of observation are detected and avoided.

By using a flask graduated at 102.4 for 60 c.c. no correction for volume of precipitated casein need be made. In no case is it necessary to heat the sample before polarising.

## A NEW PROCESS FOR THE SEPARATION OF THE OPIUM ALKALOIDS.

By PROFESSOR DR. P. C. PLUGGE.

WHILST we are in possession of accurate processes for the separation of the cinchona alkaloids, unfortunately this is not the case with those of opium, and I have therefore endeavoured to find those reagents most suitable for the separation of the latter. As the result of numerous experiments I now use for the separation of the six most important alkaloids (narcotine, papaverine, narceine, thebaine, codeine, and morphine) the following reagents: sodic acetate, potassic ferricyanide, sodic salicylate, potassic sulphocyanate, and ammonia.

(a) SEPARATION OF NARCOTINE AND PAPAVERINE FROM THE OTHERS.

To carry this out I use sodic acetate. As I have already communicated in a previous paper, strong solutions of morphine, codeine, and thebaine, are not precipitated by this reagent. Narceine is precipitated from concentrated solutions, but no longer if the percentage does not exceed .16. But papaverine is thrown down from a very weak solution (1—30,000), and narcotine from a still weaker one (1—40,000). Sodic acetate will therefore eliminate the two last alkaloids, and there is no danger of narceine co-precipitating if the fluid is only sufficiently diluted. The alkaloids precipitate as such in a pure state.

(b) SEPARATION OF THE NARCOTINE AND PAPAVERINE.

After having tried various means to separate the precipitate *a* I thought potassic ferricyanide would answer best. The two alkaloids are very difficult to separate, as they behave similarly towards most reagents, and they even do so apparently when treated with the ferricyanide. If, however, the solution contains no more than .25 per cent. or less, the narcotine is no longer precipitated, whilst papaverine is still thrown down from solutions containing 1—3,000. Even solutions containing only 1—4,000 still yield a yellowish precipitate after some time. It is therefore plain that if the precipitate obtained

by sodic acetate is dissolved in just sufficient dilute hydrochloric acid, and then diluted to such an extent as to contain no more than .25 per cent., the addition of potassic ferri-cyanide will only precipitate the papaverine. If, after standing for twenty-four hours, the liquid is filtered, the filtrate will contain all the narceine, which can be obtained as pure alkaloid by addition of ammonia. The precipitate consists of hydroferri-cyanate of papaverine, which may be weighed as such, but which for qualitative purposes is conveniently made into the pure alkaloid by digesting with a weak soda ley or dilute ammonia. To make absolutely sure, the isolated alkaloid may be redissolved in weak hydrochloric acid and once more precipitated with ammonia.

(c) SEPARATION OF NARCEINE.

After filtering off the fluid *a* we have in solution, besides the excess of sodic acetate, thebaine, codeine, and morphine, also the narceine. To recover the latter it is sufficient to concentrate the fluid on a water-bath, and when at last the liquid contains more than .16 per cent. the excess will crystallise out on allowing to cool. The precipitation is, of course, incomplete, and depends on the degree of concentration.

(d) SEPARATION OF THEBAINE.

I now make use of a strong solution of sodic salicylate, to remove the thebaine. The liquid contains, besides this alkaloid, narceine, codeine, and morphine. Thebaine salicylate is very insoluble, as solutions of thebaine containing no more than 1—2,000 are still precipitated by the reagent. The other alkaloids are not affected. After standing from twenty-four to forty-eight hours the deposit is filtered off and washed. For quantitative purposes it may be weighed as pure thebaine salicylate after drying at 100° C.; but for the purpose of testing, the pure alkaloid must be isolated by digesting the compound with weak soda ley until every trace of salicylic acid is removed.

(e) REMOVAL OF THE EXCESS OF SODIC SALICYLATE AND THE TRACES OF NARCEINE AND THEBAINE.

The filtrate from *d* contains, besides codeine and morphine, small quantities of narceine and traces of thebaine, and also excess of sodic salicylate, which must be removed before we can estimate the two first. I therefore add dilute hydrochloric acid, and after some little time filter off from the salicylic acid. The filtrate is now shaken out with chloroform, which removes the last traces of salicylic acid, also the narceine and thebaine, whilst codeine and morphine are not dissolved. I must warn against using too much hydrochloric acid, so that a little sodic acetate may still be left undecomposed. If the liquid contains free hydrochloric acid, chloroform will not properly extract the narceine and thebaine.

(f) SEPARATION OF CODEINE.

The liquid is now gently heated to expel any chloroform, and after being somewhat concentrated, mixed with a solution of potassic sulphocyanate, which throws down the codeine. Previous experiments with the hydrochlorates of morphine and codeine had shown me that even a 4 per cent. solution of the former gives no turbidity with the reagent, but the latter is readily precipitated in the cold from its .5 per cent. solution. Even solutions containing only .17 per cent. still yield small crystals of codeine hydro-sulphocyanate after being left in contact with the reagent for forty-eight hours. So



with care it is easy to practically separate the codeine, leaving all the morphine in solution.

(g) SEPARATION OF THE MORPHINE.

The filtrate from *f* is now mixed with a little ammonia, and allowed to stand in an open beaker till the excess of ammonia is practically evaporated. The greater part of the morphine crystallises out, and may then be collected. The little codeine still remaining is not precipitated. As, however, the liquid retains a not inconsiderable amount of morphine, the following process will be found more accurate for quantitative purposes, especially when working on small quantities of alkaloids.

The liquid is rendered alkaline by ammonia, and after standing at rest for some time in a separating funnel, repeatedly shaken with either chloroform or ether, which will remove the codeine, but not the morphine. The liquid is now acidified with hydrochloric acid (to redissolve the morphine), then heated to about 60°C., and repeatedly shaken out with amyl-alcohol, after addition of slight excess of ammonia.

How far the process suits quantitative analysis I wish to show by the following test analyses.

1. ESTIMATION OF NARCOTINE IN THE PRESENCE OF MORPHINE.

10 c.c. of a solution containing .1354 grm. of narcotine, and .11516 grm. of morphine, were mixed with 5 c.c. of 10 per cent. solution of sodic acetate. Precipitate weighed .1315 grm. = 97.12 per cent. narcotine recovered.

In two other experiments respectively 100 and 97.5 per cent. were obtained.

2. ESTIMATION OF NARCOTINE IN PRESENCE OF OTHER ALKALOIDS.

50 c.c., containing .28612 narcotine, .1027 morphine, .1228 codeine, and .1544 thebaine. Sodic acetate precipitated .2845 grm. of narcotine, or 99.43 per cent.

3. ESTIMATION OF PAPAVERINE IN PRESENCE OF MORPHINE AND OTHER BASES.

40 c.c. of a solution containing .28635 grm. of papaverine and .1415 of morphine yielded with sodic acetate .281 grm. = 98.15 per cent. papaverine.

50 c.c., containing .3247 papaverine, .0638 morphine, .1319 codeine, and .0915 thebaine, obtained .315 grm. papaverine = 97.02 per cent. recovered.

4. ESTIMATION OF PAPAVERINE BY PRECIPITATION AS HYDROFERRICYANATE.

.181 grm. hydrochlorate of papaverine yielded with potassic ferricyanide a precipitate weighing .193 grm. It ought to have weighed .19795 grm.; therefore 97.5 per cent. recovered.

5. ESTIMATION OF THEBAINE AS SALICYLATE.

From a mixture of this alkaloid with papaverine, morphine, and codeine, I succeeded in recovering 90 per cent. of the thebaine.

6. ESTIMATION OF CODEINE AS HYDROSULPHOCYANATE.

.35 grm. of hydrochlorate of codeine were dissolved in about 50 c.c. of water, and mixed with 5 c.c. of the reagent. After standing for twenty-four hours the precipitate was filtered off, and collected, on a weighed filter. Its weight amounted to .253 grm. corresponding with .2113 grm. of pure codeine. But after concentrating and allowing

to stand for another twenty-four hours, a further precipitate was obtained, weighing .082 grm., which brings the amount of codeine recovered, up to 97.55 per cent.

7. ESTIMATION OF CODEINE IN PRESENCE OF MORPHINE.

Care must be taken not to have the solution too strong, otherwise there is a danger of morphine hydrosulphocyanate crystallising out. .20643 grm. of codeine and .271 grm. of morphine (as hydrochlorates) were dissolved in 15 c.c. of water, and mixed with 2 c.c. of the reagent. I obtained 101.14 per cent. codeine, plainly showing contamination with morphine.

Further experiments showed it is not possible to leave all the morphine in solution without suffering a considerable deficiency of codeine. I could in fact only recover about 70 per cent. of the latter.

MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

THE DETECTION OF FAHLBERG'S SACCHARIN. C. SCHMITT. *Repert. Anal. Chemie*, 30.—To detect the presence of this substance (ortho-sulphamido-benzoic acid) in wine, the author recommends the following method:—100 c.c. of wine are acidified with sulphuric acid, and well shaken in a separating funnel, with 50 c.c. of a mixture of equal volumes of ether and petroleum spirit. The extraction is twice repeated. The mixed ethereal liquids are evaporated in a silver or porcelain dish with a few c.c. of soda ley, and when dry, the residue is heated for half an hour in the air bath up to 250° C. The fused mass is then dissolved in water and tested for *salicylic acid* by acidifying with dilute sulphuric acid, shaking with ether, evaporating the latter, and adding a drop of ferric chloride to the residue. This process is based on the fact that Fahlberg's saccharin yields sodic salicylate on fusing with sodic hydrate. The process gets more complicated when salicylic acid has been added to the wine as a preservative.

L. DE K.

TEST FOR ANILINE COLOURS IN WINES OR FRUIT JUICES. C. O. CURTMAN. *Zeitschr. f. Anal. Chemie*, H. 4.—Aniline colours are often used to colour diluted wines or artificial fruit juices, jams, etc. The author uses for their detection, Hofmann's well-known isonitрил test. 4 c.c. of wine were coloured with fuchsin, and then mixed with 4 c.c. of potash ley, and 2 drops of chloroform. After first gently warming for a minute and subsequent boiling, the characteristic smell of isonitрил was plainly perceptible. The sulpho compound of rosanilin gave the reaction only after some time. The test may be rendered more delicate, on finally adding some sulphuric acid. The small quantities of compound ethers present in the wine do not interfere with the delicacy of the test. The test was also successful with aniline blue, aniline purple, aniline violet, magenta, and ponceau red, and many yellow and green anilines.

L. DE K.

THE REFRACTOMETER IN BEER ANALYSIS. SCHWARZ. *Nordd. Brauer Ztg.*, 973.—The author gives a method for determining the original gravity of beer, based on the



following principles :—The gravity of beer is dependent on the amount of alcohol, which lowers it, and the amount of extract, which increases it. One per cent. of extract increases the gravity .00395 degree, whilst 1 per cent. of alcohol lowers it by .00161 degree. Now if any beer has a gravity of 1.02122, we will have as the first equation  $395x - 161y = 2122$ . On applying the refractometer, 1 per cent. of solid matter increases the deviation by .00150 degree, and 1 per cent. of alcohol further increases it by .00062 degree. Now if the sample shows a deviation of 1.01250, we get as second equation  $150x + 62y = 1250$ .  $x$  represents the extract,  $y$  the alcohol. The value of  $x$  and  $y$  can now be readily calculated, and from their amount the original gravity is ascertained as usual.

L. DE K.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

ESTIMATION OF ROSIN IN WAX. F. M. HORN. *Rept. Anal. Chemie*, No. 34.—A sample of fat mixture submitted to the author for analysis, apparently contained wax. On investigation it proved to have an acidity equivalent of 52, a saponification ditto of 118.5, an ether ditto of 66.5, and the relation of acidity to ether equivalent as 1 : 1.28. Pure beeswax gives the following numbers : 20, 95, 75, 3.75. Paraffin could, therefore, not be present, but there might be a mixture of stearic acid, tallow, or rosin, or only one of these bodies. When treated according to the process recommended by Benedikt and Zsigmondy, no glycerine was found, proving the absence of tallow. The low ether equivalent practically excluded stearic acid, and rosin was therefore the only probable adulterant. To qualitatively test for rosin, the author first macerated with alcohol, which dissolves the rosin and takes up but little wax. The residue left on evaporation was now treated according to the process recommended by Donath, which consists in first boiling with nitric acid, then diluting with water, and finally adding excess of ammonia, when a blood-red colour conclusively showed the presence of rosin. The fusing point and the specific gravity were as might be expected higher than those of pure wax. The percentage of rosin was now calculated from the formula—

$$100 : R = (146 - 20) : (52 - 20)$$

$$R = \frac{2300}{128} = 25.4$$

The figure 146 represents the acidity equivalent of rosin (colophony).

A very satisfactory result was obtained by testing a made-up mixture of 75 parts of wax and 25 parts of rosin.

L. DE K.

## MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

TESTING FOR INDIGO. W. LENZ. *Zeitschr. f. Anal. Chemie*. H 4.—To see whether indigo has been really used, the author tests the goods as follows: Boiling water or warm alcohol of 95 per cent. should not extract any colour. No colour should be

dissolved by boiling with saturated solutions of oxalic acid or borax. When treated with the latter, no red colour should be obtained on acidifying with hydrochloric acid, and subsequent addition of ferric chloride should not turn it blue. The goods should not be affected on boiling with a 10 per cent. solution of alum, or a 33.3 per cent. solution of ammonium molybdate. A mixture of stannous and ferric chlorides in equivalent proportions should completely destroy the colour on warming. The colour should be completely soluble in boiling glacial acetic acid. On adding to this solution, first double the volume of ether and then some water, the bulk of the colouring matter should float between the two layers. The ether should be but faintly coloured, and the acid be perfectly colourless, and remain so on adding a little hydrochloric acid. The dye should not yield any sulphuretted hydrogen on boiling with hydrochloric acid, and no smell of isonitril on heating with caustic potash and chloroform. L. DE K.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

## OLEOMARGARINE AND MICE.

To the Editor of the ANALYST.

DEAR SIR,—I was very much struck by the ingenuity of the method proposed in your last, for distinguishing between butter and butterine, that is, margarine, for which the name "mouse test" is most aptly proposed; I should be inclined to go farther, and make the method quantitative thus. Present to the mouse saucers containing pure butter and the sample for analysis; should it prefer the former, try it with a saucer of butter containing 10 per cent. margarine side by side with the sample, then one of 20 per cent., and so on, until the mouse prefers to partake of the suspected sample first; its degree of adulteration will then be ascertained within 10 per cent., which is near enough for all practical purposes. I doubt whether the taste of a mouse would be sufficient to distinguish smaller differences than this.

Cats might be employed in a precisely similar manner to rapidly analyse milk, and thus supply a widely-felt want.

Thanking you in anticipation for inserting this,—I remain, dear Sir, yours faithfully,

HARRIS, Professor and Doctor, G. A.

"Gum Tree" Office, 68, Gilpin Grove, Upper Edmonton.

N.B.—It is not advisable to put away lactometers and margarimeters together.

## BOOKS, &amp;c., RECEIVED.

AMERICAN Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; The Animal Alkaloids: Ptomaines and Leucomaines, by A. M. Brown, M.D.; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Druggist's Circular; Eighteenth Annual Report of the State Board of Health of Massachusetts; Food Adulteration and its Detection, by J. P. Ballershall, Ph.D.; Hospital Gazette; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Scientific American; Society of Arts Journal.

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"Public Analyst" would be much obliged to any of his colleagues who can give him particulars of any case that has occurred where an appointed analyst has had to enforce payment of his salary and fees against any local authority, or to resist an arbitrary reduction of the same by suing for the agreed amount. Particulars will be held in strict confidence (if so desired), and may be directed to the Editor of the ANALYST, 325, Kennington Road, London, S.E., and endorsed outside, "Reply to Public Analyst," when they will be duly forwarded.



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NOVEMBER, 1887.

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## ON REICHERT-MEISSEL'S METHOD OF BUTTER ANALYSIS AND ITS APPLICATION FOR THE EXAMINATION OF BUTTER AND BUTTER SUBSTITUTES.

BY DR. RUDOLF WOLLNY.

(Translated from the *Milch Zeitung*, 1887, Nos. 32, 33, 34, and 35.)

SINCE the separation of the fatty acids has, by Otto Hehner, been made the basis of butter analysis, the process originally proposed by Reichert and afterwards modified by Meissel, has been most generally adopted, the volatile fatty acids being separated by distillation from the other acids, and their amount determined by standard alkali.

Hehner had preferred to estimate gravimetrically the fatty acids insoluble in water, and to separate them by thorough washing with boiling water from the other substances resulting by decomposition of the butter-soap by acids. In this mode of procedure it is to be remembered that butter-fat contains, together with very readily soluble butyric acid, a small amount of fatty acids, which are soluble with more difficulty, and which therefore at first remain mixed with the insoluble acids. This portion of soluble fatty acids can only be removed by a most thorough and long-continued washing with a very large quantity of boiling water. It follows that the result is to some extent dependent upon the mode of washing and the quantity and temperature of the water employed. It may further be influenced by the mode of drying of the washed fatty acids, as by heating their weight somewhat increases by oxydation.

The method therefore yields trustworthy and concordant results only when the conditions of operation are rigorously the same, and different observers may get somewhat different results with the same sample of butter. This, as well as the lengthy washing of the fatty acids with at least one litre of boiling water, has gradually led to the more

general adoption of Reichert and Meissl's method, which has been tested by many analysts. Its reliability has been confirmed by most, and it has finally been generally adopted. Thus it has been accepted in 1885 by the Union of Bavarian Analytical Chemists, as the only method to be used. They direct that it is to be carried out as follows:—

"5 grms. of the clear-filtered butter-fat are saponified on a water-bath in a flask capable of holding 300 to 350 c.c., with 10 c.c. of a solution of pure caustic potash in 70 per cent. alcohol (20 grms. KHO in 100 c.c. alcohol). When the fat has completely dissolved, the alcohol is driven off by blowing air into the flask. The soap is then dissolved in 100 c.c. of water, decomposed with 40 c.c. of dilute sulphuric acid 1:10, and 110 c.c. are distilled off, a few small pieces of pumice having been added. 100 c.c. are filtered, and titrated with deci-normal alkali, rosolic acid or phenolphthalein being used as an indicator. The number of c.c. used is increased by  $\frac{1}{10}$ , corresponding to the total quantity of the distillate."

In accordance with these directions, the majority of butter analyses have lately been made (in Germany), because the directions of the Bavarian chemists have been the only recognised ones, being concisely and critically drawn up.

Hitherto the method has been used exclusively, as it was originally intended, for the testing of butter for admixture with foreign fats, and there has been no occasion to use it for the converse case—namely, for the detection of small quantities of butter when mixed with foreign fats—because such an investigation has so far been without object in the control of our food supply. But when the German (and other) Parliaments were considering the prohibition of butter mixtures, and the question was raised whether small quantities of butter can accurately be determined in butter substitutes, the case became of importance.

Personally, I had no hesitation to affirm the possibility of such determination, and I expressed my opinion that even 5 per cent. of butter could be accurately estimated, it being proposed in Parliament (German) that 4 per cent. be the permissible limit.

On practical trial, my expectation was by no means realised. Since 1880 I had frequently used the Reichert process, both for the analysis of mixed butters, and of mixtures made by myself, and I had always, when operating upon the same sample obtained most concordant results. I had frequently the satisfaction to be told by manufacturers that my figures closely corresponded with the proportions of butter and margarine employed. I owe it, however, chiefly to chance that I have unwillingly escaped the sources of error contained in the Reichert-Meissl method.

Other chemists have not been so fortunate—notably those who, at the request of the German margarine manufacturers, carried out a series of analyses of mixtures with the most contradictory results, and who came to the conclusion "that the Reichert-Meissl method in its present form is perfectly unsuitable for the exact determination of small quantities of butter in margarine," to quote the words of the highest analytical authority, Professor R. Fresenius.

The analysis and report of Professor Fresenius were the chief cause of the withdrawal of the clause in the Bill referring to the prohibitions of butterine mixtures. The analysis and report are as follow:—



ANALYSES OF MARGARINE MADE BY MEMBERS OF THE UNION OF MANUFACTURERS OF BUTTER-SUBSTITUTES.

Manufacturer.	Sample marked.	Composition.		Highest possible percentage of butter-fat.	Results of analyses.	Analyst.	Remarks.
		Fat.   Milk.					
		Kilos.	Litres.				
L. Lang & Sons Nürnberg.	A	100	62.4	2.18	2.4-3.0	Dr. Kaiser.	{ Two samples off one piece.
	A	100	62.4	2.18	.51	Prof. Kämmerer.	
	B	100	60	2.1	4.02	Dr. Seippel.	{ Samples B, C, and D, are off the same piece respectively.
	B	100	60	2.1	3.30	Dr. Stutzer.	
	B	100	60	2.1	1.92	Dr. J. Koenig.	
	C	100	100	3.5	3.08	"	
Eisenlohr Bros., Barmen.	C	100	100	3.5	5.20	Dr. Stutzer.	
	C	100	100	3.5	3.85	Dr. Seippel.	
	D	100	100	3.5	8.9	"	
	D	100	100	3.5	7.5	Dr. Stutzer.	
Kuntzelmann, Dresden.	D	100	100	3.5	7.11	Prof. J. Koenig.	{ Off one piece.
	E	100	60	2.1	2.86	Dr. E. Kayser.	
Mürmann & Co., Berlin.	E	100	60	2.1	6.3	Dr. Filsinger.	{ "
	F	100	49	1.8	9.12	Dr. Bein.	
	F	100	49	1.8	6.57	"	{ "
	G	100	60	2.1	3.3	Dr. Thörner.	
Müller & Co., Gildehaus.	G	100	60	2.1	2.5	Dr. P. Jeserich.	{ "
	H	100	60	2.1	1.1	"	
	H	100	60	2.1	3.6	Dr. Thörner.	{ "
	J	100	60	2.1	5.1	"	
	J	100	60	2.1	0.0	Dr. P. Jeserich.	{ "
	K	100	50	1.8	15.0	Dr. H. Süßengut.	
	K	100	50	1.8	10.5	"	{ "
	L	100	50	1.8	9.5	"	

Wiesbaden, June 7th, 1887.

Frankfurt Margarine Co.,

Bornheim, near Frankfurt.

You have requested me to report to you whether the method of Reichert and for the estimation of volatile fatty acids in butter was applicable to the accurate estimation of small quantities of butter in margarine, such as are present in common margarine. You especially requested me to ascertain the variation of the amount of volatile fatty acids in the raw materials of margarine manufacture—namely, rapeseed and earthnut oils.

You have for this purpose supplied me with twelve samples of margarine and four of oil, for the purity of which you are responsible.

I have analysed the whole of these samples in duplicate, most of the margarines, under the exact conditions prescribed by Meissl.

In most without exception comparatively wide fluctuations were exhibited, not only in different samples, but also by duplicate analyses of the same sample.

The consumption of the distillate from 5 grms., of deci-normal acid, was for each sample as follows:—

Margarine	No. 2	·24	1·34	·47 c.c.
"	No. 5	·22	·55	1·56 c.c.
"	No. 9	1·61	1·54	·44 c.c.
"	No. 12	·39	2·42	·99 c.c.
Oil	No. 2	·12	·55	
"	No. 4	·27	·67	

According to various statements to be found in chemical literature, 5 per cent. of butter-fat would consume from ·92 to 1·31 c.c. deci-normal alkali.

From these figures it is to be concluded that the method which originally was devised for the analysis of butter, and which for this gives satisfactory results, does not appear applicable to the analysis of margarine, and that only in the case of a comparatively large amount of butter, about 10 per cent., the presence of the same in margarine can be proved.

Differences, such as I have stated above, and which generally would not be very significant in a case of pure butter, are of the greatest importance in that of fats, which yield but a very small proportion of volatile fatty acids, and it appears that the method in its present form is not applicable to their analysis.

It may further be concluded, from the fact that duplicate analyses of the same sample showed fluctuations, that these are rather due to the mode of operation than to the difference in the composition of the materials.

The method might probably, after an exact study of the various possible influencing circumstances, be modified, and the *modus operandi* so far exactly specified, that the differences may be avoided, or considerably reduced, so that satisfactory results may be obtained.

But whether it will be possible to effect such modification, I am not in a position to say with certainty. There is no doubt that without such modification the Reichert-Meißl process is quite inapplicable for the exact estimation of small quantities of butter in margarine.

(Signed) DR. R. FRESSENIUS.

These results surprised me greatly, since, in my own analysis of similar samples, I had never obtained results which differed so widely. However, the facts could not be disputed, and I therefore undertook the task to ascertain the sources of error which had caused the disagreement among the results. I carried out a considerable number of estimations according to the method prescribed by the Bavarian chemists, which estimations were repeated by Dr. A. Sängner, who used the same method and the same apparatus as myself. With the sole exception that, instead of the alcoholic potash solution, always 4 c.c. of a 50 per cent. potash solution and 6 c.c. of 92 per cent. of alcohol were used, the details of that method were exactly observed. The potash solution had been made from the purest alkali, from alcohol, prepared by Kahlbaum, and on distillation with sulphuric acid yielded a small quantity of volatile acid, which, in the first series of experiments used, for every 4 c.c. ·62 c.c. demi-normal solution, in the second series ·80 c.c. These figures were subtracted from those obtained from the fats. The second column of the following table gives the figures thus corrected. In the third column are numbers showing the difference between the Reichert-Meißl results and the correct results obtained as to be described further on.



## MEISSL METHOD, DR. R. WOLLNY'S RESULTS.

Description.	C.c. deci-normal alkali		
	Used.	Corrected.	Excess beyond actual quantity.
Distilled Water	·10	—	—
100 c.c. Distilled Water + 40 c.c. $H_2SO_4$	·10	—	—
"    "    + 4KHO + 40 c.c. $H_2SO_4$	·66	—	—
"    "    "    "    "    "	·55	—	—
"    "    + 8KHO + 40 $H_2SO_4$	1·32	—	—
American Margarine, Lake	1·10	·48	·38
"    "	·88	·26	·16
"    "	·88	·26	·16
"    "	1·32	·70	·60
"    "	1·10	·48	·38
"    "	1·32	·70	·60
"    "	1·10	·48	·38
"    "	·99	·37	·27
"    "	·99	·37	·27
"    "	1·10	·48	·38
"    "	·99	·37	·27
"    "	·99	·37	·27
"    "	·99	·37	·27
"    "	1·10	·48	·38
"    "    Progress	1·21	·59	·49
"    "	1·32	·70	·60
French Margarine, Foudoir	1·32	·70	·60
"    "	1·55	·92	·82
Vienna Margarine, Siebenschein	1·43	·81	·71
"    "	1·10	·48	·38
Pure Butter, Holstein	29·59	28·97	·32
"    "	30·14	29·52	·87
"    "	30·36	29·74	1·09
"    "	30·03	29·41	·76
"    "	29·59	28·93	·28
Butter and Margarine mixed	1·43	·81	·71
"    "	1·65	1·03	·93
"    "	1·54	·92	·82
"    "	1·76	1·14	1·04
"    "	1·54	·92	·82
Butter mixture 1 (2 per cent.)	2·20	1·58	·98
"    1 (2 " )	2·09	1·47	·87
"    2 (4 " )	2·53	1·91	·66
"    2 (4 " )	2·75	2·13	·88
"    3 (6 " )	3·30	2·68	·87
"    3 (6 " )	3·41	2·79	·98
"    4 (8 " )	3·74	3·12	·72
"    4 (8 " )	3·96	3·34	·94
"    5 (10 " )	4·51	3·89	·98
"    5 (10 " )	4·40	3·78	·87
"    6 (50 " )	16·50	15·88	1·57
"    6 (50 " )	15·84	15·22	·91
"    7 (85 " )	25·96	25·34	1·03

C.c. deci-normal alkali

No.	Description.	C.c. deci-normal alkali		Excess beyond actual quantity.
		Used.	Corrected.	
49	Butter mixture 7 (85 per cent.)	35.63	25.01	.70
50	" 8 (90 " )	26.73	26.11	.27
51	" 8 (90 " )	27.17	26.55	.71
52	" 9 (95 " )	28.60	27.98	.84
53	" 9 (95 " )	28.38	29.76	.62
MEISSL METHOD, DR. A. SANGER'S RESULTS.				
54	100 c.c. distilled water	.20	—	—
55	" " + HO c.c. H <sub>2</sub> SO <sub>4</sub>	.20	—	—
56	" " + 4KHO + 40H <sub>2</sub> SO <sub>4</sub>	.88	—	—
57	" " " " " "	.77	—	—
58	" " +8 " +40 "	1.54	—	—
59	Sesame Oil	1.87	1.07	.97
60	" "	1.43	.63	.53
61	Earthnut Oil	2.09	1.29	1.19
62	" "	1.98	1.18	1.08
63	" "	.99	.19	.09
64	Cotton oil	1.54	.74	.64
65	" "	1.54	.74	.64
66	Pure butter, Holstein	29.81	29.01	.36
67	" "	31.13	30.33	1.68
68	" "	31.35	30.55	1.90
69	" "	30.47	29.67	1.02
70	" "	30.47	29.67	1.02
71	" "	28.27	27.47	1.18*
72	" "	30.91	30.11	1.46
73	" "	30.81	30.00	1.35
74	" "	29.15	28.35	.30*
75	" "	32.23	31.43	2.78
76	Butter and Margarine, mixed	1.54	.74	.64
77	" "	1.32	.52	.42
78	" "	.99	.19	.09
79	" "	1.21	.41	.31
80	" "	1.10	.30	.20
81	Butter mixture 1 (2 per cent.)	2.31	1.51	.91
82	" 1 (2 " )	1.87	1.07	.47
83	" 2 (4 " )	2.86	2.06	.81
84	" 2 (4 " )	2.64	1.84	.59
85	" 3 (6 " )	4.40	3.60	1.79
86	" 3 (6 " )	3.19	2.39	.58
87	" 4 (8 " )	3.63	2.83	.43
88	" 4 (8 " )	3.85	3.05	.65
89	" 5 (10 " )	4.18	3.38	.47
90	" 5 (10 " )	3.96	3.16	.25
91	" 6 (50 " )	10.06	15.26	.95
92	" 6 (50 " )	15.84	15.04	.73
93	" 7 (85 " )	26.07	25.27	.96
94	" 7 (85 " )	25.74	24.99	.63
95	" 8 (90 " )	26.84	26.04	.20
96	" 8 (90 " )	27.18	26.48	.64
97	" 9 (95 " )	29.04	28.24	1.10
98	" 9 (95 " )	28.60	27.80	.66



The above 98 experiments fully confirm the results of other analysts and of Professor Fresenius, and show that considerable variations (in some cases upward of 1 c.c.) occur in the analysis of the same sample. For instance, in experiment 85 the mixture containing 6 per cent. of butter required 3.60 c.c., and in No. 90 containing 10 per cent. of butter, only 3.16 c.c. The figures with one and the same sample of pure butter varied from 28.93 to 31.43, a difference corresponding to nearly 10 per cent. With the materials used in the manufacture of margarine, and a mixture of six samples of margarine, and nine samples of oil, the fluctuations amounted from .19 to 1.29 c.c., corresponding to about 4 per cent. of butter.

I therefore likewise conclude that the method in its present form is really quite useless for the purpose which has been stated. In order to obtain correct results the sources of error had to be ascertained.

Inasmuch as the fluctuations of the results did not exhibit any regularity whatever, which might have given a clue to the explanation, and as every possible care had been taken to prevent the absorption of acid or alkaline gases by the reagents and vessels, carbonic acid was suspected to be the cause of the error, phenol-phthaline, which had been used as an indicator, being very sensitive to that body, as is well known by the researches of Warder, Thompson, and others. By blowing air through dilute phenol-phthaline solution, which has been rendered red by a drop of soda solution, the colour is destroyed, and several c.c. of deci-normal solution may be necessary to reproduce the colour. If for titration barium hydrate be used the totality of the absorbed carbonic acid is measured, but with potash or soda solution only one half, bicarbonates being neutral to phenol-phthaline.

The mode of procedure, as prescribed by the Bavarian chemists, affords plenty of opportunity for the absorption of carbonic acid. It may be derived from the gases of combination which may enter the flask, and it is surely absorbed during the blowing-in of air for the removal of the least quantity of alcohol.

Experiment 99.—4 c.c. KHO and 6 c.c. alcohol were evaporated to dryness on a water-bath, with frequent blowing-in of air. The soap taken up with 100 c.c. of distilled water and distilled with 40 c.c. of  $\text{H}_2\text{SO}_4$  required 2.2 c.c. deci-solution.

Experiment 100.—In a similar experiment, without blowing-in of air, 2.2 c.c. were likewise required, whilst 4 c.c. of KHO directly distilled with water, and  $\text{H}_2\text{SO}_4$  required .8 c.c.

This absorption of  $\text{CO}_2$  has hitherto been overlooked in the Reichert Meissl process, and although, as will be seen further on, it is not the only source of error, it is in itself sufficient to explain the difference in the results of the analysis made on behalf of the margarine manufacturers, quoted above; it is the sole cause of the results in the case of margarine and oil made by Prof. Fresenius. The volatile acids found by him in these materials are almost solely carbonic acid, after the exclusion of which 5 grms. of margarine, sesame, and earth-nut oil yield less volatile acids than correspond to 1 c.c. deci-normal solution.

The error thus caused is an entirely uncontrollable one, dependent upon a great variety of circumstances. It may amount to as much as 10 per cent. of butter, and renders the results obtained by the method quite inaccurate.

The following experiment will show that the error is indeed due to carbonic acid, and not to acetic acid formed by the oxidation of alcohol, as has been suggested by O. Schweissinger (*Pharm. contrib.*, 1877, p. 320).

Experiment 101.—100 c.c. alcohol were boiled with 4 c.c. KHO solution under a reflux condenser for one hour in a closed flask, and the alcohol was then distilled off. The residue, dissolved in 100 c.c. of water and distilled with  $H_2SO_4$  as before, required 1.6 c.c. deci-solution, whilst 4 c.c. of the same potash solution directly distilled with  $H_2SO_4$  without alcohol consumed 1.2 c.c., and 100 c.c. alcohol required .4 c.c.

In order to get rid of this source of error, I attempted to choose an indicator which should be sensitive to butyric and other soluble fatty acids, but unaffected by carbonic acid. I have tried litmus, resolic acid, methyl-orange, and cochineal, with a solution of fatty acids obtained by distillation of butter, with a solution of carbonic acid in water, and with a mixture of both. It was found that while litmus and rosolic acid were almost equally sensitive to carbonic and fatty acids, methyl-orange and cochineal were far too delicate with both.

Having thus failed in the choice of a new indicator, I endeavoured to find a means to entirely prevent the absorption of carbonic acid. The purest kinds of caustic potash which I could obtain all furnished acid distillates corresponding for 2 grms. to .6—1.2 c.c. deci-normal solution. Potassium carbonate was found to be sufficiently soluble in alcohol to affect the results. A freshly-prepared alcoholic potash solution required .6 c.c., and after twenty days, during which time it had become dark brown, and had deposited some crystals, still consumed .6 c.c. when distilled as described. I therefore entirely abandoned the use of caustic potash, and replaced it by a 50 per cent. solution of caustic soda, in which chloride, sulphate, nitrate, and carbonate of soda are quite insoluble, so that if caustic soda is dissolved in its own weight of distilled water it leaves a crystalline sediment of these salts, the clear solution, after separation from the sediment, being pure and almost free from carbonic acid. Even if such a solution attracts carbonic acid from the air, the carbonate formed separates again and deposits as crystals. A soda solution thus prepared may be kept ready for use, a constant allowance being made for the very small amount of volatile acids which it contains.

(To be continued.)

#### CONTRIBUTION FROM THE LABORATORY, 11, BILLITER SQUARE.

##### NOTE ON A SHORTENED FORM OF CERTAIN FACTORS.

By H. DROOP RICHMOND.

MANY factors which are commonly used in calculating analytical results may be shortened by the use of various simple fractions.

For example, to convert  $BaSO_4$  into  $SO_3$  it is necessary to multiply by .34335 which equals (within .005 per cent.)  $.343333 = .333333... + .01$ , or  $\frac{1}{3} + \frac{1}{100}$ .

In other words, divide the weight (X) of  $BaSO_4$  first by 3, and then by 100, and add the two together.

To convert  $Mg_2P_2O_7$  into  $MgO$ , multiply by .4, and subtract  $\frac{1}{100}$  of that from it, and then add  $\frac{1}{1000}$  of the last.



Thus:—

·3265 grms.  $\text{BaSO}_4 = \cdot 1088 + \cdot 0033 = \cdot 1121$  grms.  $\text{SO}_3$ .

·4726 grms.  $\text{Mg}_2\text{P}_2\text{O}_7 = \cdot 1890 - \cdot 0189 = \cdot 1701 + \cdot 0002 = \cdot 1703$  grms.  $\text{MgO}$ .

TABLE OF FACTORS THUS SHORTENED.

Subst. weighed.	Subtract required.	Factor.	Shortened Factor.	Error.
$\text{BaSO}_4$	$\text{SO}_3$	·34335	$\frac{1}{3} + \frac{1}{180}$	·005 p. c. —
$\text{BaSO}_4$	S	·1373	$\frac{1}{4} - \frac{1}{180}$ , or wt. of $\text{SO}_3 \times \cdot 4$	—
$\text{Mg}_2\text{P}_2\text{O}_7$	$\text{MgO}$	·36036	$\cdot 4 - \cdot 04 + \cdot 0004 - \cdot 00004$	—
$\text{AgCl}$	Cl	·2473	$\frac{1}{4} - \frac{1}{160}$	·01 p. c. +
$\text{SiO}_2$	Si	·4667	$\frac{1}{2} - \frac{1}{30}$	—
c.c. N Acid	<div style="display: inline-block; vertical-align: middle;">           { Nitrogenous            { Organic substances         </div>	·0875	$\frac{1}{10} - \frac{1}{30}$	—

## REVIEWS.

FOOD ADULTERATION AND ITS DETECTION: WITH PHOTOMICROGRAPHIC PLATES AND A BIBLIOGRAPHICAL APPENDIX. By JESSE P. BATTERSHALL, Ph.D., F.C.S., Chemist, U.S. Laboratory, New York City. E. and F. N. Spon, London and New York.

THIS addition to the continually increasing mass of literature on the subject of food analysis comes to us with two special claims on our attention, because it is not only the first actual book dealing with such matters that has been written by an American Author, but it is also the work of the official chemist in the government laboratory. It therefore would, we presume, occupy a similar standing in America to that of Mr. Bell's work in this country. After a short introduction on the general subject of adulteration, and the defects of the law in the States and elsewhere, we are given a table of the sophistications actually existing in practice at the present time. From this chapter we gather that the author's views, although very lenient on certain points (as for instance on the subject of oleomargarine), are yet stronger on some others than we are at present accustomed to in this country. What, for example, would the advocates of the "pure beer" bills, and the brewers, respectively, say to the following list of adulterants of our national beverage?—"artificial glucose and bitters, sodium bicarbonate, salt." The book commences with tea, and then takes up coffee, cocoa, milk, butter, cheese, flour and bread, sugar, honey, confectionery, beer, wine, liquors, water, vinegar, pickles, olive oil, mustard, pepper, spices, and a few miscellaneous matters. There is nothing novel about the processes, and, indeed, the author distinctly states in his preface that "many of the methods of examination given are quoted in a condensed form from the more extensive works on food-analysis." So far as we have seen, this remark might with justice be amended, as they say in parliament, by omitting the word "many" and substituting the words "nearly all." The whole diction of the work is however short, and to the point and there is a commendable absence of mere padding. The processes are clearly expressed with quite sufficient detail, and here and there the author shows great facility of explanation in a few words. This is especially noticeable in the short and lucid explanation of the nature of polarised light given as an introduction to the use of the polariscope. It is in the illustrations that the book makes its great bid for popularity, and here it must be admitted that nothing like it has as yet appeared. They are chiefly microphoto-

graphs and are beautifully reproduced, the pictures of some of the starches under polarised light being especially interesting.

There is also a commendably complete bibliography at the end of the book, and a full digest of the laws against adulteration. Like most of the higher priced American books, the printing, paper and general get up are all excellent, and we have no doubt that the work will command a good market. There are several tables of analysis of standard samples resulting from the author's own work, those under "tea" being specially full and instructive. The following list of so-called "standards" compiled from the book will be interesting to analysts, as showing how nearly the views held in America approach to our own in many cases.

Tea. { Soluble ash not under 40 per cent. of the whole ash.  
 Insoluble leaf not over 60 per cent.  
 Total ash not under 4.5 per cent. and not over 7 per cent.

Coffee.—Density of 10 per cent., infusion and following formula:  $C = \frac{1.00 (1.020 - D)}{12}$

Milk.—12 per cent. milk solids and 3 per cent. of fat.

Butter. { Koettstorfer's method—1 grm. butter takes 221 m. grms. KOH.  
 Hehner's method—87.5 per cent. insoluble acids.  
 Reichert's method—2.5 grms. take 13 c.c. decinormal KOH.

Alum in bread.—Deduct 0.013 from the weight of the precipitate found.

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MANUAL FOR THE PHYSIOLOGICAL LABORATORY. By VINCENT DORMER HARRIS, M.D. Lond., F.R.C.P., and D'ARCY POWER, M.A., M.B., Oxon, F.R.C.S. Fourth edition, with illustrations. London: Bailliere, Tindall and Cox.

WHEN this book was first published, we had occasion to welcome it as a most desirable addition to medical educational literature, and we then predicted a prosperous future and many fresh editions. Events have justified this forecast, and we now have the fourth addition before us. The authors have wisely seen the necessity of not standing still, and the present book comes forward with a considerable increase of matter of at once the most modern and most useful type. In that part of the work in which we, as chemists, are specially interested, namely, the portion devoted to physiological chemistry, the improvement is exceedingly noticeable, the information given being uniformly in accordance with the latest views, and we should say that any medical student honestly reading it up could not fail to get all he wants for his examinations. The great feature of the book is its purely practical character, the student being taught entirely by experiments, first in histology, with the microscope, then in physiological chemistry, with his tubes and reagents, and finally he is taken into practical physiology and trained in the use of all the modern instruments, such as the sphygmograph, the cardiograph, the ophthalmoscope, the laryngoscope etc. Although there is scarcely an unnecessary word to be found in its pages, it is yet by no means a mere cram book and, in our opinion, the authors deserve the gratitude of all students for the interesting way in which a subject, generally dreaded by them as complicated and difficult, is so fully placed before their minds. The publishers have also done their part of the work well, both paper and type being excellent, while the unusually strong binding supplied will enable the book to stand the wear and tear of the laboratory.

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A SHORT MANUAL OF ANALYTICAL CHEMISTRY; QUALITATIVE AND QUANTITATIVE, INORGANIC AND ORGANIC. By JOHN MUTER, Ph.D., M.A., F.R.S.E., F.I.C. etc. London: Bailliere, Tindall and Cox, and Simpkin and Marshall. Philadelphia, P. Blakiston Son and Co. Third Edition, illustrated.

It would be manifestly impossible to criticise this book in the ANALYST, because the critic would always be suspected of favouring a work by the editor of the journal for which he is employed. It is therefore only permissible to give the headings of the chapters, which will convey some idea of the scope of the book.

Part I. Qualitative analysis.

- Chap. I. The processes employed by practical chemists
- " II. Detection and separation of metals.
- " III. Detection and separation of acids (inorganic and organic).
- " IV. Detection of unknown salts, simple and complex.
- " V. Detection of alkaloids and of poisons in mixtures.

Part II. Quantitative analysis.

- Chap. VI. Volumetric analysis, use of the nitrometer and colorimetric analysis.
- " VII. Gravimetric analysis of metals, acids, etc.
- " VIII. Ultimate organic analysis.
- " IX. Weighing and measuring, specific gravity and vapour density.
- " X. Analysis of water, air and food:
- " XI. Analysis of drugs, urine and urinary calculi.
- " XII. Analysis of gases, polarisation, spectrum analysis and melting points.

The moderate price of the work (6s. 6d.) brings it within the reach of all students. Several of the courses are original, and only to be found in this book, being the outcome of the author's twenty years experience in the teaching of practical chemistry.

THE ANIMAL ALKALOIDS: CADVERIC AND VITAL, PTOMAINES AND LEUCOMAINES, CHEMICALLY, PHYSIOLOGICALLY AND PATHOLOGICALLY CONSIDERED, IN RELATION TO SCIENTIFIC MEDICINE. By A. M. BROWN, M.D. With an Introduction by Professor ARMAND GAUTIER. London: Bailliere, Tindall and Cox.

It was a happy thought on the part of Dr. Brown to collect in one volume all the scattered facts and researches on this subject, and were the work only equal to the conception all would be well. We do not for a moment pretend to judge of the medical part of the book, nor do we wish to enter into any discussion on the merits or demerits of the author's theory of the causation of disease, although, as chemists, our instincts would naturally lie in the direction of the views supported in this work. What lies strictly within our province is the chemical part of the book, and, as will be seen from the remarks that follow, we fear we cannot speak of it so favourably as we could wish. Every one admits the eminence of Professor Gautier, but, as his own nation say, *toujours perdrix* is apt to induce a feeling of weariness. One rises from the perusal of the book with too much of the feeling that there is but one Gautier and that Brown is his prophet. When a man presumes to write a purely chemical section in a book he should first be sure that he has mastered the nomenclature of the science. Nothing is more irritating than to meet with the same substance under two different names in the very same page, and in this book we continually find such an occurrence. The author speaks within six lines of a "platinichloride" and of a "chloroplatinate," meaning always the same body. He continually talks of sulphurated hydrogen, but still be sometimes deigns to drop into the humble sulphuretted hydrogen. Again, what do our readers think of this specimen of nomenclature, and of a nicely conceived equation:—"heat splits up choline into glycole and trimethylamine,  $C_3 H_8 NO = C_2 H O_3 + CHN$ ."

Here is also a beautifully lucid sentence:—"Its aldehydic constitution explains, as Professor Gautier has shown, that it reduces the melange of ferricyanide, ferro chloride." We are sorry to show our ignorance, but we must confess that we have yet to learn what hydrate of trimethylhydroxethylenine-ammonium is. One more specimen, and we have done. After directing us to *alkalise* an acid liquid with chalk, the process proceeds thus:—"In order to neutralise the liquor of distillation it is evaporated nearly to dryness, and the ammonia sulphate removed, treating the remaining mother-liquor with pure concentrated alcohol; this dissolves the alkaloid sulphate, and leaves the ammoniacal sulphate." For real perspicuity this can, we think, hardly be surpassed. In a word, the whole chemical part of the book constitutes a grand moral lesson on the subject of the cobbler and his last. Let Dr. Brown get a friend who is a scientific chemist to revise this chapter in his next edition, and then he will have produced a really interesting work.

EXERCISES IN QUANTITATIVE ANALYSIS, WITH A SHORT TREATISE ON GAS ANALYSIS. By W. DITTMAR, LL.D. (Edin.), F.R.S., F.R.S.E. Glasgow: William Hodge and Co.

As might be expected from the high position of the author as a practical chemist and teacher, this work is an excellent one for advanced students. It would, in our opinion, be specially suitable for men preparing to undertake commercial work, and for examinations of the Institute of Chemistry. In the preface the author calls attention to the section on the analysis of gases, and he is well justified in doing so, because this portion of the book is specially lucid and complete. The author states that he believes there is no work dealing comprehensively with this subject except the well-known treatise of Bunsen, but here he has evidently overlooked Mr. Sutton's book on volumetric analysis, which also contains a large and well-written section devoted to the analysis of gases. The work commences with a minute description of manipulative details, and the author apologises for going so deeply into this groundwork. He confesses that, had he been criticising his own book some ten years ago, he would have objected to such detail, but he says that his experience in teaching has taught him differently. On this subject Dr. Dittmar's words are worth quoting, he says:—

"The technicalities of quantitative analysis are the very things which the student is not likely to find out by himself. He had better be drilled into doing them correctly. What is the good, for instance, of letting him spoil a series of ammonia determinations by mismanaging his chloro-platinate precipitates? It surely is better to show him quite directly what he has to do, and if it is, why should not the book tell him, and thus save the time of the teacher? No fear of any talented student being spoiled by a course of judicious drilling. It is just he that must be made alive to the fact that no amount of scientific knowledge will enable a man to get through a quantitative analysis successfully unless he has the "canning" as well as the knowing, and unless he attends to all those little practical details which to him at first sight may appear to be irrelevant. Cheerful and conscientious devotion to all the protracted drudgery that may be involved in one's duty is certainly a lesson worth learning, and it is one of the educational functions of quantitative analysis to inculcate the lesson. And as to the talented student's weaker brother? Why, he *must* be drilled, or else he may learn nothing at all.

"Of course, here, as everywhere, we must beware of extremes, and take care not to disgust the student with his work. It is as well, even at the earlier stages, to occasionally break the monotony of analytical work by the interpolation of an exercise in preparative chemistry. With students who have already been "broken in," a capital plan is to give them unnamed substances and let them *find* the exercise or set of exercises that they are meant to work, taking care not to give them any help except where it may become necessary to prevent sheer waste of time."

All these remarks are exceedingly good and to the point, but we must be excused in saying that if books were to be made so full of elaborated details as to be practically self-teaching, there would then be no use for teachers of practical chemistry. Our view is that a book should lay down the principles of the methods, and that the practical



teacher should personally fill in the minute details, and duly impress on his students the necessity of rigorous attention to such matters. It must not, however, be supposed that because we disagree with over detail in a practical book, we in any way depreciate the value of the work before us, because we think that the author has been needlessly sensitive in defending himself from possible attack, and that the book does not show so very strongly in this respect when one comes to read it, as would be expected from the preface. In conclusion, we have only to express our conviction, from a most careful perusal, that this is an excellent book, and that a student who has worked through it will be turned out a good practical analyst, capable of facing real commercial work, as distinguished from mere college laboratory training.

THE NEW RELIGIO MEDICI: CHAPTERS ON PRESENT DAY SUBJECTS. By FREDERICK ROBINSON, M.D.

THIS is a book quite outside of our scope, and we must leave the handling of it to the journals dealing with religious and literary matters. It is chiefly a plea, on medical and other grounds, for a revival and abbreviation of the present services of the Church of England.

THE STUDENT'S HANDBOOK OF CHEMISTRY, WITH TABLES AND CHEMICAL CALCULATIONS. By H. LEICESTER GREVILLE, F.I.C., F.C.S. Second Edition. Edinburgh: E. and S. Livingstone.

THE new edition of Mr. Greville's book comes before us with increased matter, but the size has, if anything, been reduced by adopting a smaller type. It is a small book of 88 pages, about the size of "Roscoe," but containing a good deal more detail, especially in the chemical theory part, and also having analytical tables. We do not view the matter as an unmixed advantage, because we think that it is always more convenient for the student to have separate books for theoretical study at home and for practical work in the laboratory. Every one of the numerous small books on chemistry with which the market is deluged must be supposed to have some distinctive feature, and the strong point of this one is in giving full explanations and examples of calculations and of arranging the preparation and characters of the various groups of bodies in tabular form, so as to be readily assimilable. Seeing that it is a second edition the author might have been a little more careful about the revision of the text. For example, on page 25 we are told that the weight of a litre of H is 11.19 litres, but all the calculations which follow are made on 11.16. Again, on page 35 we find this equation:  $-H_2SO_4 + Zn = SO_4 + H_2$ —an awkward misprint. Worse than this is an equation on page 200, showing copper dissolving in hydrochloric acid with evolution of hydrogen. It is a pity that an otherwise really well-conceived book should be thus occasionally disfigured.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

ESTIMATION OF PROTEIN IN MILK. R. PALM. Zeitschr f. Anal. Chemie H 3.—The author prefers the process of Millon and Commaille when slightly modified. This process consists in adding a solution of mercuric nitrate, which throws down a compound of protein and mercuric oxide. As precipitation proceeds, nitric acid is gradually liberated and partly redissolves the compound. The author therefore advises to, from time to time, neutralise the liquid with weak potash ley, when the precipitation will be complete. A slight excess of potash is not hurtful, but rather beneficial, as one may then be certain, only mercuric oxide precipitates, and not a basic nitrate. An excess of uncombined mercuric oxide will be in the precipitate, but this is of no consequence when

the following plan is adhered to. The precipitate is collected on a weighed filter and thoroughly washed with alcohol, dried at 100° C. and weighed. The filter plus contents is then treated with *aqua regia*, and from this solution the mercury is thrown down as sulphide, which is weighed and calculated to oxide. This deducted from the first weight gives the amount of protein. Or the moist precipitate of protein plus mercuric oxide is digested with baric hydrate, which dissolves out the protein. The filtrate is cautiously neutralised with dilute sulphuric acid to remove barium, and after filtering it is evaporated to dryness and the protein weighed.

Another method is based on the property of tannic acid, of forming an insoluble compound with protein, even in very weak solutions (1—100,000), but there was no proper method to separate the tannic acid from the precipitate. Alcohol has been tried, and it certainly completely extracts the tannin, but a considerable amount of protein also passes into solution. If, however, instead of alcohol, anhydrous ether is used, the last trace of tannin is removed without the least loss of protein. The only condition is the absolute dryness of the precipitate. The ether must on no account contain water, though a little alcohol does not hurt; in fact, we may conveniently use a mixture of three parts of ether and one part of absolute alcohol. The precipitate may also be heated to 35° C. with excess of solution of plumbic acetate. Plumbic tannate is formed and the protein passes in solution. A current of  $H_2S$  is now passed to remove the lead and the filtrate is evaporated to dryness. The protein thus liberated is dried at 100° C. and weighed.

L. DE K.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

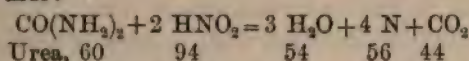
REACTIONS OF KAIRINE, ANTIPYRINE AND ANTIFEBRIN. KOHN. Jour. d'Alsace-Lorr.  
*Kairine*, with a drop of ferric chloride in a weak aqueous solution, instantly assumes a violet colour, that rapidly passes to brown. An excess of ferric chloride to a strong solution of kairine produces an almost black precipitate. Bichromate of potassium, in neutral solution, gives an intense coloration and separates a violet pigment on standing, which, dissolved in alcohol, forms a black solution. *Antipyrine*, in weak solution, forms a rose colour with ferric chloride that is visible in a 1 to 100,000 solution. With nitrous acid added to a dilute solution a blue-green colour is produced, while in the concentrated solution, green crystals are deposited. *Antifebrin*, with the reagents previously mentioned, undergoes no change, but boiled with potassium hydrate, evidence of the existence of aniline is obtained and, after distillation, potassium acetate may be found in the retort. In the *Apoth. Zeitung* the following test is given for antifebrin: Boil a few centigrammes of antifebrin with 1 c.c. of officinal solution of potassium hydrate, and hold suspended in the tube a glass rod which has been dipped in a solution of chlorinated lime; the drop of solution suspended on the end of the rod will acquire an amber colour, which, on continuing the ebullition, passes little by little to violet. This violet coloration results from aniline, which is produced from the antifebrin by the boiling caustic potash. It may be well to further note that if the test is made directly with aniline the violet coloration of the drop appears at once, without the primary change to amber colour, as in the case of antifebrin.

J. W. E. (*Am. Journ. Pharm.*)

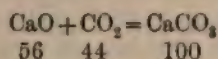


# LY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

METHOD FOR THE VOLUMETRIC ESTIMATION OF UREA. DR. G. CAMPARI. *Chim.*, 1887, page 156.—This method is based upon the decomposing action by nitrous acid upon urea, to form carbonic anhydride, nitrogen gas, and water. In a flask, of about 200 c.c. capacity, place 20 c.c. of a 10 per cent. solution of potassium, then 2 c.c. of the urine, or of the liquid containing the urea, and 10 c.c. of a 5 per cent. solution of sulphuric acid (or 1 c.c. of the diluted sulphuric acid of U. S. P., 1880). After the addition of the acid through the safety tube, the evolved gases through the other tube alongside, descending into a flask containing 10 c.c. of lime-water. Warm very slightly the urea-containing flask, so that it soon takes at least fifteen minutes time. When the connecting-tube becomes moist with hot aqueous vapour, the operation is at an end, and the lime solution should be added at once. Now measure 10 c.c. of this liquid, turbid with its suspended carbonate and colour rose-violet, with a drop of an alcoholic solution of phenolphthalein, and, first, determine the number of c.c. of a solution of oxalic acid, containing 0.0165 gram to the litre, that are needed to neutralise the 10 c.c. of the liquid. Then measure the volume of the oxalic acid solution used by 0.0165 and subtract the product from 0.15; the difference indicates the amount of urea contained in the 10 c.c. of the urine, or the urea-containing liquid examined. Pavesi and Rotondi have found that 1 c.c. of lime-water is neutralised by 0.00241 gram of tartaric acid; therefore every 10 c.c. of lime-water should contain 0.001273 gram of CaO, and 110 c.c. of lime-water should then contain 0.14003 gram of CaO, corresponding to 0.15 gram of urea. The chemical reactions of this new method are as follows, first:—



andly:—



since 44 grms. of  $\text{CO}_2$  are equivalent to, or neutralise, 56 grms. CaO, then 100 grms. of CaO are equivalent to 60 grms. of  $\text{CO}(\text{NH}_2)_2$ , for 60 grms. of  $\text{CO}(\text{NH}_2)_2$  gives off 44 grms. of  $\text{CO}_2$  (as seen in the first equation); and if 56 grms. of CaO equal or are equivalent to 60 grms. of  $\text{CO}(\text{NH}_2)_2$ , then 0.14003 gram of CaO (the amount contained in 110 c.c. of lime-water employed) equals 0.15 gram of urea, thus:—

$$56 : 60 :: 0.14003 : 0.15.$$

and if you employ 110 c.c. of lime-water, and if, after the operation, you subtract from the product the number 0.15, the quantity of urea corresponding to the lime not precipitated in the 110 c.c. of lime-water, you will have the quantity of urea corresponding to the lime precipitated. In order to know the quantity of urea corresponding to the lime not precipitated (which remains in solution), take from the product, that one c.c. of oxalic acid neutralises one molecule of CaO, and this corresponds to one molecule of urea. Then, if 1000 c.c. of the above oxalic acid corresponds to 1.5 gram of urea, 1 c.c. corresponds to 0.0015 gram of urea. Multiply the number of the c.c. of the solution of oxalic acid used (to

neutralise the 10 c.c.) by 0·0015, which equals, equivalently, the quantity of urea in 1 c.c., or, which is the same thing, multiply that number by 0·0165 for 11 times the proportion (this will give the urea corresponding to the lime left in solution), and subtract this quantity of urea from 0·15 (urea corresponding to the lime contained in 110 c.c. of lime-water); the difference represents the urea corresponding to the lime precipitated and contained in the liquid analysed.

Results of a number of determinations are given, first, upon a solution of urea, 25 to 1000, and then upon urine, in comparison with the well-known process of Liebig; the latter results are as follows:—

Urine used.	Oxalic Acid used for 10 c.c. of Lime-water.	Urea in 1 litre, (Campari.)	Urea in 1 litre, (Liebig.)
2 c.c.	6·6 c.c.	20·55 grms.	20·20 grms.
2 "	6·3 "	23·05 "	22·90 "
2 "	6·45 "	21·80 "	21·55 "
2 "	6·95 "	17·70 "	17·35 "

These results, according to the author, demonstrate that this nitrous acid method conduces to the best results obtainable. But, in order to secure absolutely accurate results, it is necessary that the heating of the flask be slow, in order to avoid, especially, the raising of the vapour of nitric acid that could be formed by the decomposition with the sulphuric acid, from the potassium nitrate, always present in the commercial nitrite.

In the rule previously given, there are directed 110 c.c. of lime-water rather than 100, in order to facilitate analytical calculations. 110 c.c. of lime-water contains 0·14003 grm. of CaO, that corresponds to 0·15 of urea; this number is easy to remember, while the same cannot be said for the number 0·1363 (urea) corresponding to the lime (1·1272) contained in 100 c.c. of lime-water.

In conclusion, the subjoined equivalent table is given, in which is stated the quantity of urea contained in a litre of any possible sample to be examined, indicated by the number of cubic centimetres of the oxalic acid solution used to neutralise 10 c.c. of the lime-water.

Oxalic Acid used, (c.c.)	Urea, (per litre.)	Oxalic Acid used, (c.c.)	Urea, (per litre.)	Oxalic Acid used, (c.c.)	Urea, (per litre.)	Oxalic Acid used, (c.c.)	Urea, (per litre.)
4·0	42·00	5·0	33·75	6·0	25·50	7·0	17·25
4·1	41·00	5·1	32·95	6·1	24·70	7·1	16·45
4·2	40·35	5·2	32·10	6·2	23·85	7·2	15·60
4·3	39·55	5·3	31·30	6·3	23·05	7·3	14·80
4·4	38·70	5·4	30·45	6·4	22·20	7·4	13·95
4·5	37·90	5·5	29·65	6·5	21·40	7·5	13·15
4·6	37·05	5·6	28·80	6·6	20·55	7·6	12·30
4·7	36·25	5·7	28·00	6·7	19·75	7·7	11·50
4·8	35·40	5·8	27·15	6·8	18·90	7·8	10·65
4·9	34·60	5·9	26·35	6·9	18·10	7·9	9·85

J. W. E. (*Am. Journ. Pharm.*)



ON SOAP ANALYSIS. F. M. HORN. Zeitschr. f. d. Chem. Ind. H. 16.—The usual plan of testing soap for sodium chloride, sulphate or carbonate, by first dissolving the soap in alcohol and analysing the residue, does not give correct results, as the salts are far from being insoluble in commercial alcohol. This is caused by the alcohol containing water, and matters are made still worse if the soap contains (as it often does) from 20 to 60 per cent. of moisture. The author therefore proposes to estimate salt by dissolving the soap in water and adding sufficient dilute nitric acid to completely separate the fatty and resinous acids. The chlorine is then estimated in the filtrate by nitrate of silver. To estimate any sodium (or potassium) carbonate, the soap is first dried at 20—40° C., afterwards at 110—120° C. The dried mass is then exhausted with absolute alcohol and the residue dissolved in water and titrated. The author thinks it still better to make a direct estimation of CO<sub>2</sub> in the original soap. Sulphates are estimated in a portion of the insoluble matter, or may be taken in the ash.

L. DE K.

ALBUMEN IN URINE, ITS VARIOUS FORMS AND THEIR TESTS. DR. T. GRAINGER STEWART. Quart. Comp. Med. Science.—The forms of albumen met with in the urine are:—

(1). *Serum Albumen*, a substance which, according to Hammarsten, constitutes 4.516 per cent. of the blood serum. It is almost constantly present in urine which contains any variety of albumen. Although a less diffusible body than serum globulin, it is capable of passing through membrane. (2). *Serum Globulin or Paraglobulin*, the globulin of the blood serum, of which it constitutes 3.103 per cent. It is met with in almost all albuminous urines, its proportion to the serum albumen varying in different instances. (3). *Peptone*, a product of gastric and pancreatic digestion of albuminous substances, also occurring in the process of transformation of tissues and of inflammatory effusions. It is a readily diffusible substance, occasionally met with in the urine in association with or apart from serum albumen. (4). *Propeptone, or Parapeptone, or Hemialbumose*, a substance or group of substances intermediate between albumen and peptone, constituting a stage or stages of transformation from the one to the other. It is highly diffusible, and is occasionally met with in the urine under conditions corresponding to those under which peptone occurs. (5). *Acid Albumen, or Syntonin*, one of the derived proteids obtained by the action of acids upon albumen. It is easily produced artificially by the addition of acid to albuminous urine, but may occur naturally in certain cases. (6). *Alkali Albumen*, another derived proteid, produced by the action of alkalies upon albumen. It is readily produced artificially, but is also found naturally in the urine. (7). *Hæmoglobin*, the combination of hæmatin and globulin naturally existing in the red corpuscles of the blood. It sometimes appears in the urine, particularly in cases of hæmaturia and hæmoglobinuria, also in certain septic conditions, and after inhalation of arseniuretted hydrogen, transfusion of blood, and otherwise. (8). *Fibrin*, a proteid substance which does not normally exist as such in the blood. It is met with in the urine in hæmaturia, in some cases of chyluria, and in certain varieties of renal casts. (9). *Mucin*, the chief constituent of mucus, is a derived proteid substance. It frequently becomes superadded to the urine after secretion, and may be derived from any part of

the urinary tract. (10). *Lardacein, Waxy or Amyloid Material*, familiarly known as a pathological substance within the body, is said to be occasionally demonstrable in renal casts.

Of these ten varieties the last four are of little practical importance—mucin alone being indeed worthy of special comment, and that mainly because of the difficulties which its presence raises in regard to the reliability of certain tests for serum albumen.

The following are the tests for the albumens, put in tabular form.

TABLE SHOWING TESTS FOR THE CHIEF FORMS OF ALBUMEN.

	SERUM ALBUMEN.	SERUM GLOBULIN.	PEPTONES.	PROPEPTONES.	ACID AL- BUMEN.	ALKALI ALBUMEN.
Heat.						0
Heat with nitric acid.	Opacity.	Opacity.	0	0	0	Opacity.
Heat with acetic acid.						
Cold, nitric acid.	Opacity.	Opacity.	0	Opacity dissolved by heat.	Opacity.	Opacity.
Metaphosphoric acid.	Opacity.	Opacity.	Opacity diminished or dissolved by heat.	Opacity diminished or dissolved by heat.	0	Opacity.
Acidulated brine.	Opacity.	Opacity.	Opacity diminished or dissolved by heat.	Opacity diminished or dissolved by heat.	Opacity.	Opacity.
Picric acid.	Opacity.	Opacity.	Opacity dissolved by heat.	Opacity dissolved by heat.	Opacity.	Opacity.
Potassio-mercuric iodide.	Opacity.	Opacity.	Opacity dissolved by heat.	Opacity dissolved by heat.	Opacity.	Opacity.
Potassium ferro-cyanide.	Opacity.	Opacity.	0	Opacity dissolved by heat.	Opacity.	Opacity.
Dilution with water.	0	Slight opacity.	0	0	0	0
Magnesium sulph.	0	Opacity.	0	0	Opacity.	Opacity.
Fehling's solution.	Brownish-red or mauve	—	Rose pink or purple.	Rose pink or purple.		
Randolph's test.	—	—	Yellow opacity.	Yellow opacity.		

W. H. D.

TESTS FOR A FEW OXYANTHRAQUINONES. E. NOACK. LIEBIG'S ANNALEN, 240.—

Xanthopurpurine is easily soluble in baryta water with brownish-yellow colour; anthracrysone is not. If xanthopurpurine is gently heated with a small lump of caustic potash and a few drops of water, it passes into purpurine. First of all, it forms potassium xanthopurpurine of a yellowish-red colour, and on adding water we get an alkaline solution of purpurine of a pure red colour. If the xanthopurpurine is absolutely pure, the strongly diluted alkaline solution soon decolourises by the combined influence of air and sunlight. If the alkaline solution is acidified, purpurine is precipitated. .01 grm. still shows the reaction. Of tetraoxyanthraquinone there are two varieties. The first, which is soluble in benzole, gives the following reactions: strong sulphuric acid gives a brownish-yellow, alkalis an emerald-green colour. These colours do not readily change. The second variety is insoluble in benzole and gives with sulphuric acid a violet, and with alkalis an emerald-green, which colours, however, soon fade. Pentaoxyanthro-



inone is scarcely soluble in benzole and dissolves in strong alkalies with a green, in weak alkalies with a brownish-yellow colour. Dimethylantracrynone is insoluble in benzole and dissolves in sulphuric acid with a fuchsia colour, in alkalies and ammonia with a reddish-yellow colour. Methylantracragallol, from *O* toluylic acid, and the same from the *P* acid, are easily soluble in alcohol, whilst rufigallic acid is not. Their barium salts are insoluble. Hot ammonia dissolves with a blue, alkalies with a violet or green colour. Sulphuric acid gives a red, which changes into green by nitric acid. L. DE K.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

## SOMERSET HOUSE AND MILK ANALYSIS.

To the Editor of the ANALYST.

SIR,—My attention has been called to a communication from Mr. Estcourt, published in THE ANALYST for September, with respect to an unusual disparity between the respective results, obtained by him and in this department, in the analysis of what purported to be portions of the same sample of milk.

Mr. Estcourt has tried hard to make your readers believe that the extraordinary difference between the results arose from the alleged uncertainty of the amount of decomposition which takes place in milk which has been kept for a certain period, but your readers will probably be somewhat surprised to hear that, instead of establishing a case against the practicability of making a reliable analysis of a sour milk, he has rather tempted and, in my opinion, successfully, to prove that the portion of the milk submitted to us for analysis had either been seriously tampered with, or that it was not the same milk as that analysed by him.

The reference samples of milk which are submitted to us consist, almost invariably, of the third portions of the original purchase retained by the inspector, and the bottles, which are always full, or nearly so, are sealed with the official seal of the local authority. In the case of the sample in question the seal on the bottle bore the letter E, in old English, and the milk, which in bulk did not represent more than a third of the capacity of the bottle, appeared more like a residue than the portion usually retained by the inspector.

In this case, as in that of every sample of milk referred to us by the justices, we determined the ash and chlorine, and the proportions of those constituents alone, which could not be affected by any change in the milk, proved beyond question that the sample submitted to us contained at least 40 per cent. of added water, the amount which we stated in our certificate. Further, so far from the sample affording proof of undue decomposition, its condition showed that less than the average amount of decomposition had occurred, and, moreover, the amount of fat which practically remains unchanged in kept milk, also supported our conclusion.

I give the results of our analysis, which are consistent throughout, and which indisputably establish the accuracy of our conclusions:—

Non-fatty solids	..	..	4.79 per cent.
Fat	..	..	1.47 "
Ash	..	..	.47 "
Chlorine	..	..	.06 "

These results speak for themselves and require no further comment from me.

I am, Sir, yours faithfully,

J. BELL.

The Laboratory, Somerset House,  
11th October, 1887.

## ANIMATED REAGENTS.

*To the Editor of the ANALYST.*

SIR,—Observing the very interesting letters from American and English sources on the use of animals as lactometers and margarimeters, I thought well to write you and show that not to your country alone does the honour of such brilliant researches belong, but that we in Germany are not behind. In the *Pharm. Centralh.*, 1887, p. 361, you will find the question asked—"Has saccharin a sweet taste to animals?"—and the answer given in the following paragraph, which I have translated.

"Has saccharin a sweet taste to animals? So much stir has been made about sulphanimo benzoic acid, that the following will be important to our readers. If a only partly-covered basin is filled with solution of arsenic, and is exposed, it is soon filled with many corpses of small insects, and also of ants. The animals must have been attracted to it by some means unknown to scientists. A solution of saccharin when exposed is, however, not visited by insects, in fact they seem to dislike it. Then, a fly-paper, covered one side with syrupus simplex, and the other with solution of saccharin, was frequented by ants, who, however, only tasted the true syrup, and carefully avoided the saccharin."

Look, Sir, at the splendid test here shadowed forth for syrups made with saccharin. As Harris, the well known and always-to-be-respected G.A., so sensibly pointed out in your last paper, there would be some difficulty in keeping the lactometric cats and margarimetric mice together. But this is where our German discovery excels, because the saccharometric ants would not interfere with the other instruments. The only precaution necessary in employing these useful insects would be for the analyst to tie up securely the lower end of his trouser legs.—Yours, etc., NARETHOR, A.S.S.

[We have several other jocular letters on this subject, but must ask our correspondents to excuse their non-insertion, as enough fun has already been poked at the matter.—ED. ANALYST.]

## CARBOLISED PEAT.

*To the Editor of the ANALYST.*

DEAR SIR,—In common, no doubt, with the majority of your readers, I was very much interested in the communication on Commercial Carbolic Compounds in your last number. But my object in writing is to point out what appears to me to be a serious objection to the specification proposed for carbolic powders: the specification describes the powder as "having for its base any siliceous or other inert mineral matter with which the acid will not combine." The word *mineral* in the above practically excludes peat.

I am afraid it would require a very elastic imagination to consider peat a mineral.

At the present time, when so much carbonised peat is used in the London districts, and with such a markedly superior effect, as shown by the greatly diminished number of cases of fever in the districts using it, it is important that it be not excluded through the adoption of a form of tender that specifies mineral bases only.

Of the inertness of peat towards carbolic acid there can be no question, and of its superiority over silica or siliceous residues I think there can hardly be two opinions.

Carbolised peat is very largely manufactured now from compact brown peat, free from unchanged vegetable fibre, perfectly stable, and very active as an absorbent.

I should suggest that the word "mineral" be struck out of the proposed specification.—I am, dear Sir, Yours very truly,

J. WEST KNIGHT, F.I.C., etc.

The Cambridge Laboratory, Oct. 20th, 1887.

Public Analyst for Cambridge and District.

## APPOINTMENT.

MR. ALFRED ASHBY has been appointed Medical Officer of Health, Public Analyst and Gas Examiner for the Borough of Reading.

## BOOKS, &amp;c., RECEIVED.

AMERICAN Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Bench Book for the Test Tube Work in Chemistry, by H. T. Lilley, M.A.; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Druggist's Circular; Exercises in Quantitative Analysis, with a Short Treatise on Gas Analysis, by W. Dittmar, LL.D.; F.R.S.; Hospital Gazette; The Illustrated Sidney News; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Scientific American; Society of Arts Journal.

## NOTICE TO CORRESPONDENTS.

ALL Communications on Literary or Exchange Matters to be sent to 325, Kennington Road, London, S.E.



# THE ANALYST.

DECEMBER, 1887.

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(All Abstracts deferred till next month owing to pressure of Original Matter.)

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting was held at Burlington House on Wednesday, the 9th ult., the President, Mr. A. H. Allen, in the chair.

The minutes of the country meeting were read and confirmed.

On examining the ballot papers the scrutineers reported that the following gentlemen were elected:—As members, Mr. H. Smith, M.D., F.I.C., F.C.S., etc., Public Analyst for Woolwich and Plumstead; J. M. Vargas, analytical chemist, Bogota. As associates, G. W. Gray, assistant to Mr. Norman Tate; W. Chattaway, assistant to Mr. Allen.

The following papers were read and discussed:—

“Allowances for Decomposed Milk.” By A. W. Stokes.

“On the Preservation of Milk Samples.” By Otto Hehner.

“The Substitution of Asbestos Cloth for Blotting Paper, in Mr. Adams' process of Milk Analysis.”

“Does Neutral or Sub-Acetate of Lead precipitate Hop Bitters?” By W. Johnstone.

“On Filtration.” By Otto Hehner and Henry D. Richmond, who exhibited the filter and apparatus employed.

“Alumina as a Natural Constituent of Wheat Flour.” By W. C. Young.

Mr. HEHNER read the correspondence from Mr. Estcourt and Dr. Bell, which has recently appeared in THE ANALYST, on the subject of milk analysis, and also the further communication from Mr. Estcourt which appears on another page.

The three last-named papers, by Messrs. Johnstone, Hehner, and Young, will appear in the next issue of THE ANALYST.

## ANALYSIS OF DECOMPOSED MILK.

C. ESTCOURT, F.I.C., F.C.S.

THE communication made by me upon the above subject, which appeared in the September number of the ANALYST (page 168), seems to have caused considerable trouble to Dr. Bell, the gentleman who represents the Somerset House Laboratory.

Dr. Bell, in face of such a series of analyses as I presented, feeling himself upon the horns of a dilemma, is compelled, apparently, to take one of two courses: he must either admit (what is already recognised by the majority of public analysts) that the analysis of curdled, partially decomposed milk is unreliable, or he must take the unusual and extraordinary course adopted by him in his communication of October 11th (*vide* ANALYST, page 221).

Dr. Bell says, "Mr. Estcourt has tried hard," etc., when, in fact, I confined myself to a bare statement.

Dr. Bell thinks it right to suggest that "the sample had been seriously tampered with," rather than disbelieve in the unreliability of a necessarily fallible method.

The fact is, his figures are only explicable upon the supposition that no complete mixture is possible of fat, curd, and whey after long standing.

If the case I have given stood alone, it might be deemed impossible that such a result could be obtained as was obtained by the Somerset House analysts. It is, however, not unusual for these gentlemen to differ both as to results and inferences with public analysts. The Somerset House chemists reported upon the analysis of a milk contained in a bottle which had been broken in transit, and from which some of the liquid portion had escaped. This was in connection with a certificate of adulteration given by Mr. E. W. T. Jones, the result being that the case was dismissed, together with another which depended upon the Somerset House certificate (see ANALYST, vol. i., 1877, page 74).

It is interesting to observe in the second case alluded to, that Somerset House found 0.14 per cent. ash more than Mr. Jones had done, and advanced the ash result as affording the main proof of the genuine character of the milk, (see also difference of 0.10 per cent. between Somerset House and Dr. Hill, ANALYST, vol. i., 1877, page 40).

Regarding my own case, and assuming that the ash given by Somerset House is correct, I will compare it with milks analysed by Mr. E. W. T. Jones and Mr. Percy Smith (see ANALYST, vol. i., page 76, and vol. v., page 149). In these communications may be found several examples of very low ash, thus:—

	Solids not Fat.	Ash.
E. W. T. Jones' sample	8.97	0.56
" "	7.96	0.53
" "	7.67	0.49
C. Estcourt's	7.70	0.47
Percy Smith's	9.10	0.49

In these analyses, unfortunately for this reference, the chlorine is not estimated, but I do not doubt that, if estimated, the results would have borne out the views of the authorities upon the subject.

Mr. John Pattinson (ANALYST, vol. i., page 98) says, "Usually the amount of chlorine is from .06 to .08 per cent."

Mr. Otto Hehner (ANALYST, vol. vii., pages 5 and 6) says, "The percentage of ash found by Somerset House (in a referred milk case of his) cannot be correct." He also (same communication) shows solids not fat reduced by decomposition in  $7\frac{1}{2}$  weeks from 8.37 to 4.22 per cent. Dr. P. Vieth (ANALYST, vol. vii., page 215) gives loss of solids not fat ranging from 2.0 to 2.66 per cent in four days.



There is also a case in which Dr. Wynter Blyth analysed a milk, a portion of which, when sour, being analysed by the Somerset House chemists, gave the following results:—

	Solids not Fat.	Fat.
Wynter Blyth	8.81	2.09
Somerset House	7.84	3.35

(See ANALYST, vol. ii., page 202.) Dr. Blyth reported it skimmed; Somerset House certified to added water. Here is an error in fat almost equal to their error in solids or fat in my case.

In face of these facts and a large number which have never been published, it will be a matter of regret to all public analysts that Dr. Bell should prefer to consider many of them incompetent and some officials dishonest, rather than admit that the inferences from the analysis of decomposed milks are sometimes unreliable.

NOTE.—The sample to which Dr. Bell's letter refers was obtained from a farmer's man at a railway-station in town, and was therefore not divided into three parts. I divided it myself, sealing and returning one part, with the certificate, to the inspector. The magistrates themselves forwarded it to Somerset House at the farmer's request. The farmer had been supplied by our Inspector with a duplicate of my original sample.

It appears from Dr. Bell's letter he had never before seen a sample sealed with an analyst's own seal, as provided by the Act when a sample has been procured in transit.

It is also rather late in the day to put forward figures of ash and chlorine high, if of any value (which is, I submit, doubtful in the case of decomposed milk), ought to have figured upon the Somerset House certificate.

The sample was sent to Somerset House on the 25th March, but the certificate was not received until three weeks later, a total period of nearly eight weeks from the date when it was first procured.

The PRESIDENT said in reference to the division of the sample that Mr. Estcourt had further explained that it was divided at the station into two parts, one part being given to the farmer, and the other taken to Mr. Estcourt, who again divided his part into two, sealed up one portion, and used the other for the analysis. On finding it adulterated he returned the sealed-up portion to the inspector, who produced it in Court in the usual way. The part given to the farmer was analysed on behalf of the defendant by a local chemist. The only point of importance was that Dr. Bell suggested as the ash of his sample was so very low it could not have been the same milk as was analysed by Mr. Estcourt. But it did not appear that Dr. Bell gave the ash and chlorine in his certificate, although he states in his letter that he always makes these determinations; it would be interesting to know why he suppressed the figures. Of course he could understand that observations might sometimes be made for the sake of learning, and not for reporting, but that would not account for Dr. Bell reporting the chlorine and ash in certain cases, and not in all.

With respect to the ash, it was curiously low. In Dr. Bell's book there was an analysis of genuine milk given as low as .65 per cent., and with 14 per cent. of water it could be reduced to .56 per cent. Mr. Estcourt had shown by a number of examples that the error of analysis in the determination of the ash at Somerset House sometimes amounted to fully .10 per cent., and therefore the difference was perfectly capable of explanation without the insinuation made in Dr. Bell's letter.

Mr. J. Pattinson had shown that the amount of chlorine in milk was usually between .06 and .08 per cent., so that there was nothing in Dr. Bell's figures to show that the milk analysed by Somerset House was not the same milk as that analysed by Mr. Estcourt.

## ALLOWANCES FOR DECOMPOSED MILK.

By A. W. STOKES, F.C.S., F.I.C.

*(Read at Meeting, November 9th, 1887.)*

VARIOUS isolated analyses of the same samples of milk, before and after the commencement of decomposition, have been published, though, so far as I know, no series of such analyses has hitherto been printed. Even such analyses as have been recorded have usually been by different analysts, the one doing the analysis of the fresh sample, the other when decomposed. In such cases the method of analysis has not been necessarily, or even probably, identical, so that the results are not strictly comparable.

During the last few months I have made a series of analyses, using the same method for the fresh and the stale samples; this I now bring before you.

The method used was as follows:—A careful mixture of the sample was made; 5 grms. of this were weighed into flat-bottomed dishes of 3 inches diameter. After complete drying in the water-bath the total solids were weighed. The dishes were now repeatedly filled with light benzoline, boiled, dried, and re-weighed. The fat was thus taken by difference. In the case of the same milks when decomposed the 5 grms. taken for analysis were carefully neutralised by decinormal soda solution before drying; in calculating the results  $\cdot 0022$  gm. for each c.c. of  $\text{NaHO}$  was subtracted from the total solids, as recommended by Mr. James Bell, of Somerset House, in his book on "Analysis and Adulteration of Foods." All were done in duplicate. This method was not adopted as being the very best for milk analysis, but because of its simplicity, and of its near resemblance to the method quoted in the above book. Whatever error there may be in the method from the abstraction of fat possibly not being absolute, etc., runs through the series both of fresh and stale milks, but does not vitiate the comparison of these with one another. Almost all of the samples are adulterated; they are, therefore, such as usually appear in reference cases. They are actual samples sent to me by various Vestries for analysis. In most cases the vendors were fined for adulteration.

The times of keeping of the samples varied from 8 to 117 days, and the season from July 6th to November 3rd, 1887; hence a fairly wide range of changes of temperature has been tried.

In the following list are recorded the total solids, fat, and solids not fat; then under the heading of "s.n.f. + allowance" is placed the s.n.f. of the previous column, with the addition for keeping suggested by Mr. J. Bell in above book: that is, for 7 days,  $\cdot 24$ ; for 14 days,  $\cdot 34$ ;  $21 = \cdot 41$ ,  $28 = \cdot 48$ ,  $35 = \cdot 55$ , and  $\cdot 01$  more for every day after is to be added to the s.n.f. found. In the next column is placed the difference between this last figure and the s.n.f. obtained from the fresh milk. If, in spite of the allowance, the weight is less than at first, the letter "L" for "loss" is placed before the difference shown; if it is greater, the letter "G" for "gain" is prefixed, and the time during which the sample has been kept follows. In the next column I have placed the total acidity, calculated as lactic acid. In the last column, at the suggestion of our President, Mr. A. H. Allen, I have placed the difference between the original total solids and the total solids of the stale milk *plus* the Somerset House addition for decomposition. All figures are percentages. There are 26 samples; all were analysed at two periods, and three of them at three periods. They are generally arranged according to the length of keeping, beginning at the shortest time.



Sample.	Date of analysis.	Tot. sols.	Fat.	S.n.f.	S.n.f. + allowance.	S.n.f. difference.	Lactic acid.	Tot. sols. difference.
1	Sept. 8	6.90	2.06	4.84				
	" 12	6.92	2.02	4.90	5.15	G. 0.31 in 8 days	0.52	G. 0.27
2	Sept. 27	11.26	3.06	8.20				
	Oct. 12	11.26	3.28	7.98	8.32	G. 0.12 in 14 days	0.52	G. 0.24
	Nov. 3	10.72	3.10	7.62	8.18	L. 0.02 in 36 days	0.99	G. 0.02
3	Sept. 27	10.92	2.88	8.04				
Heated 66 hrs.	Oct. 12	10.90	3.10	7.80	8.14	G. 0.10 in 14 days	0.55	G. 0.32
" 168 "	Nov. 3	10.40	3.14	7.26	7.82	L. 0.22 in 36 days	1.78	G. 0.04
4	Sept. 22	10.54	2.34	8.20				
	Oct. 11	10.02	2.38	7.64	8.02	L. 0.18 in 18 days	0.61	L. 0.16
	Nov. 2	8.40	1.74	6.66	7.26	L. 0.94 in 40 days	1.09	L. 1.54
5	Sept. 22	11.13	2.44	8.69				
Heated 60 hrs.	Oct. 11	10.14	2.06	8.08	8.46	L. 0.23 in 18 days	0.36	L. 0.61
6	Aug. 18	10.23	2.83	7.40				
	Sept. 14	8.54	2.90	5.64	6.10	L. 1.30 in 26 days	1.76	L. 1.23
7	Aug. 18	10.30	2.74	7.56				
	Sept. 16	7.82	2.66	5.16	5.64	L. 1.92 in 28 days	0.97	L. 2.00
8	Aug. 29	9.72	2.14	7.58				
	Sept. 30	8.72	2.20	6.52	7.03	L. 0.55 in 31 days	0.72	L. 0.49
9	Aug. 29	10.67	2.65	8.02				
	Oct. 1	10.32	2.70	7.62	8.14	G. 0.12 in 32 days	0.77	G. 0.17
10	Aug. 5	10.96	2.82	8.14				
	Sept. 10	9.30	3.32	5.98	6.53	L. 1.61 in 35 days	1.18	L. 1.11
11	Aug. 5	9.63	2.13	7.50				
	Sept. 10	7.54	2.12	5.42	5.97	L. 1.53 in 35 days	2.07	L. 1.54
12	Aug. 12	10.45	0.14	10.31				
	Sept. 20	10.12	0.52	9.60	10.18	L. 0.13 in 38 days	1.08	G. 0.25
13	Aug. 22	9.09	2.70	6.39				
	Sept. 30	8.42	2.54	5.88	6.46	G. 0.07 in 38 days	0.82	L. 0.09
14	Aug. 10	9.93	1.92	8.01				
	Sept. 20	9.24	1.42	7.82	8.41	G. 0.40 in 39 days	1.02	L. 0.10
15	Aug. 22	9.02	2.54	6.48				
	Oct. 1	8.26	2.52	5.74	6.33	L. 0.15 in 39 days	1.18	L. 0.17
16	Sept. 20	12.76	3.28	9.48				
	Oct. 31	12.02	3.62	8.40	9.00	L. 0.48 in 40 days	0.82	L. 0.14
17	Sept. 12	12.02	3.14	8.88				
	Oct. 31	11.76	4.46	7.30	7.98	L. 0.90 in 48 days	2.12	G. 0.42
18	July 13	9.74	2.44	7.30				
	Sept. 13	9.50	2.72	6.78	7.59	G. 0.29 in 61 days	2.10	G. 0.57
19	July 12	8.12	1.92	6.20				
Smell very bad.	Sept. 14	3.68	1.44	2.24	3.07	L. 3.13 in 63 days	0.10	L. 3.61
20	July 21	12.74	4.22	8.52				
	Sept. 28	12.36	4.42	7.94	8.82	G. 0.30 in 68 days	1.24	G. 0.50
21	July 21	8.40	1.81	6.59				
	Sept. 28	7.52	2.00	5.52	6.40	L. 0.19 in 68 days	1.15	0.00
22	July 21	9.69	2.32	7.37				
Smell & Colour very bad.	Sept. 28	6.22	2.14	4.08	4.96	L. 2.41 in 68 days	0.52	L. 2.59
23	July 20	10.13	2.01	8.12				
	Sept. 28	8.80	2.10	6.70	7.59	L. 0.53 in 69 days	1.35	L. 0.54
24	July 19	8.25	2.09	6.16				
	Sept. 28	7.24	2.36	4.88	5.78	L. 0.38 in 70 days	2.01	L. 0.11
25	July 6	10.21	2.25	7.96				
	Nov. 1	9.40	1.98	7.42	8.79	G. 0.83 in 117 days	2.34	G. 0.56
26	Sept. 20	10.79	3.07	7.72				
	Oct. 19	10.54	3.39	7.15	7.63	L. 0.09 in 28 days		G. 0.22

Examining the more noteworthy of these we find that in Sample 1 there has been practically no change during the 8 days of its keeping. This is the only case where the analysis shows (by error of experiment) a very slightly higher s.n.f. after keeping than when fresh. Here, therefore, the addition of an allowance must result in a gain, as shown.

Samples 2 and 3 are milks of the same date, and of very similar constitution. They were analysed when fresh, after 14, and after 36 days. Sample 2 was left all the time at the average temperature of the laboratory (about 58° F.), but sample 3, during the latter part of the first 14 days, was kept at a temperature of 70° F. for 66 hours. During the 14 days both samples altered so little, that making the allowance for keeping, we get s.n.f. greater than in the original samples. The heating of sample 3 for 66 hours has apparently not made any appreciable difference. Both samples, after keeping 36 days, show a loss of s.n.f. after the addition of the allowance. But No. 3 received also an additional heating to 70° F. for 102 more hours; adding the allowance to this, its s.n.f. show a greater loss than in the unheated sample. The lactic acid is about twice as great in this.

In these two cases it is evident that for a short period the allowance is too great, but for a longer time it becomes too small, and that heating the sample increases the deficit—that is, hurries on the decomposition.

In samples 4 and 5 the same effect of heating is seen, though at no period is there a gain.

The alternations of the allowance being either too little or not enough are shown in the following samples, till we come to No. 19. This sample after 63 days' keeping developed an unusually disgusting odour, quite unlike that of every other sample (except one) kept either for longer or shorter periods. In it we find, after making the allowance, a loss of s.n.f. = 3.13. With this is a lactic acid the lowest of the whole series. That the mere period of keeping has nothing to do with this anomalous result is seen by reference to the samples adjoining it, Nos. 18 and 20. These, kept through about the same period and at the same temperature, actually show a gain instead of this enormous loss when the allowance is made.

No. 22 is another sample possessing after 68 days the same dreadful smell, combined also in this case with a strong yellow colour. Here, when the allowance is made a loss of s.n.f. = 2.41 is shown, and yet it has an acidity as small as that of a sample kept only for 8 days. Each side of it are milks of the same date that show but a trifling loss after making the allowance. So remarkable are samples Nos. 19 and 22 that they were repeated 8 times with concordant results.

Sample 7 shows the great loss of 1.92 s.n.f. (after making the allowance) in only 38 days, yet it has a very low acidity, and gave no very bad smell. In No. 25 is seen a sample kept for 117 days, in which, if the allowance is made there is actually a gain of s.n.f. = 0.83, while the acidity has run up very high.

For the original analysis of No. 26 I am indebted to Dr. Thomas Stevenson, this being the only sample in which I did not make the analysis while fresh. The method of our analyses being, I believe, the same I have inserted this example in the series. It shows, except in one other case, the closest approximation between the analyses of the fresh and stale samples, after making the allowance.



Reviewing the entire series by comparison of s.n.f., we find that after making the Somerset House allowance, there are 9 cases in which the allowance is too great, and 20 in which it is too small. The average gain of s.n.f.+allowance being 0.28, and the average loss 0.84. They range from a gain of 0.83 to a loss of 3.13. According to Mr. J. Bell, the s.n.f. after making the allowance ought not to differ from the original s.n.f. more than 0.10 per cent. Only 4 of these cases fell within or upon this limit.

Considering the series from the point of view of total solids only, there are 12 cases in which, after making the allowance, there is a gain shown, and 16 in which the allowance is not great enough. The average gain of total solids is 0.29, the average loss 1.00 when the allowance is made. They range from a gain of 0.57 to a loss of 3.61. In 8 cases the loss or gain shown by the s.n.f. is inverted by the total solids when the allowance is applied to each; these inversions, however, do not happen in any of the very large differences. These inversions are just what one would expect if the constituents of a milk do not decompose in an invariable manner.

The allowance is founded upon the decomposition producing principally lactic acid; and so far as this is normally produced the allowance somewhat follows with it. But the series shows that lactic acid is not by any means always the chief product of decomposition, the very worst decomposed milks (*e.g.*, Nos. 19 and 22) having but little acidity; hence the allowance breaks down. Nor does the time of keeping produce either the same difference of s.n.f., or the same amount of lactic acid even when the temperature and the quality of the milks are about the same. Increase of temperature usually increases the loss of s.n.f., and adds to the lactic acid, but not invariably. What seems to me is at the foundation of the different changes that undoubtedly take place in milks similarly treated is the nature of the initial fermentation set up. It entirely depends upon what germs reach the sample as to the change that will ensue.

Two samples, under like conditions, but differently impregnated with fungus-spores, will produce different results, and no possible allowance can be made for this, seeing that we do not know the nature of the original fertilisation. This is complicated, too, by our ignorance of the temperature at which the sample was kept before it reached us in the ordinary reference cases.

I have examined these samples microscopically, and have been surprised to find how differently they have become infected with fungus. There are mainly two classes:—those (the ropy samples) containing large-celled spores, and a thick, branching mycelium; and the ordinary samples, in which only excessively minute bacteria are seen, and no mycelium is visible. Others show both of these.

That milks do not decompose in any regular manner is obvious to the senses. Some retain their fresh appearance for a long time, while others of the same date separate rapidly into layers of serum and casein; a few burst their bottles, while others do not. If the decomposition be the same in all these cases why are the physical results so varied?

I have looked through the records of other examples of milk samples analysed at different periods by other analysts, and I find it impossible to make any allowance to count for decomposition.

A few by Dr. W. Blyth will be found in the ANALYST, vol. iii., p. 230, and others are scattered through the vols. of the ANALYST.

A notable example is shown in the September, 1887, number of the ANALYST, where, through applying an allowance, the Somerset House chemists find 40 per cent. of added water, after 27 days, while the sample when fresh contained only 14 per cent. of added water. Had any of the above samples found their way to Somerset House, it is not pleasant to contemplate the results that might have issued.

I have not worked out the serious differences of "added water" that would be indicated in the series given, but any one can easily do that. The only conclusion I can come to is that—*no allowance for decomposition can possibly be made in our present state of knowledge.* I might perhaps be allowed to suggest that in cases where stale milk is analysed some such report as follows might be made if the sample fall below the standard: "The sample was too far decomposed to enable me to form an accurate judgment of its original state, but I am of opinion that it contains *about* . . . per cent. of added water." Of course, if the sample come above the standard (without making any allowance) it may certainly be returned as genuine.

To Mr. E. Michael I owe thanks for assistance and care in carrying out much of the work here recorded.

#### DISCUSSION.

The PRESIDENT said they would agree with him that the subject brought before the Society by Mr. Stokes was one of the highest interest to them as public analysts. It was unfortunately one of the great drawbacks to the proper performance of their duties that, after having effectually and thoroughly satisfied themselves of the character of a milk, they should be liable to see justice annulled, and have their reputations slurred by reports made by the Somerset House chemists on the results of analyses of milk, sometimes four, six, and seven weeks old at least, and in which the opinion is based upon the unwarrantable assumption that milk changed on keeping at a practically uniform rate, no matter at what temperature it was kept, or whether a preservative had been added. Common-sense sufficed to discredit such an extraordinary theory, and most public analysts had within their personal knowledge cases which absolutely disproved the assumption, but Mr. Stokes was the first to bring an extensive and systematic series of observations before the Society. By the courtesy of Mr. Stokes, he (the President) had had an opportunity of seeing the paper previously, and had suggested that it was undesirable to base the proof of the rate of change on the solids not fat, as such a course left a loophole for Dr. Bell to urge that the variations were due to imperfect and unequal extraction of the fat. As Dr. Bell alleged that no change in the proportion of fat was produced by keeping the milk, it was clear that any diminution of the total solids was caused by change in the solids not fat, and hence the determination of the total solids gave at once a measure of the change which had occurred in the milk, and enabled one to avoid any but the very simplest manipulation—namely, that involved in drying the total milk-solids to a constant weight. The determinations of total solids were all made by Mr. Stokes in duplicate, and some in triplicate, so that the accuracy of his figures was beyond question, and it was not possible to evade the conclusions deducible from them by suggesting manipulative error.

From the column in Mr. Stokes' table showing the departure from the truth resulting from the analysis of the decomposed milk, and the use of the Somerset House "allowance for change," it appeared that only in five instances out of twenty-nine did the total solids thus corrected come as near as 0.1 per cent. to the actual amount found



in the fresh milk. Hence the odds were nearly six to one against Somerset House arriving at a reasonably correct result by the analysis of a decomposed milk. It was true that the figures showed they were twice as likely to find too high a result as too low, but such variations proved that the method of allowance was intrinsically worthless and false in principle, and could not be amended by a revision of the figures alleged to represent the daily variations.

He, the President, had been so much struck with the deductions to be made from Mr. Stokes' figures that he had been at the trouble of comparing the rate of change observed in cases in which the figures had been published, and the results might be worth recording in a tabular form.

Analyst concerned.	Reference.	Age of decomposed milk.	Error in total Solids after applying S. H. allowance.
			Per cent.
Wynter Blyth	ANALYST, iii., 231	21	+0.23
"	"	14	+0.66
"	"	34	+0.49
"	"	18	-2.20
"	"	153	-1.46
"	"	153	+0.48
"	"	80	-2.25
E. W. T. Jones	ANALYST, i., 74	80	-0.01
O. Hehner	ANALYST, vii., 5	{ 34	-0.19
A. Hill	ANALYST, i., 40	{ 53 (f)	-3.08
C. Estcourt	ANALYST, xiii., 168	{ 21	+0.31
J. Baynes	Communicated	{ 14	+0.37
A. H. Allen and M. A. } Adams	Communicated	{ 27	-4.07
M. A. Adams	Communicated	{ 27	+0.55
"	"	{ 28	-0.53
"	"	{ 15	-2.05
"	"	{ 33	-5.05
"	"	{ 42	+0.79
"	"	{ 47	+0.53
"	"	{ 47	+0.79
"	"	{ 47	+0.37
"	"	{ 47	+0.35
P. Vieth	ANALYST, vii., 215	{ 4	-0.88
		{ 4	-1.80

With reference to the case in the table in which Mr. Adams and the President were jointly concerned, they both analysed the milk when fresh with closely concordant result. After an interval of a fortnight the speaker analysed his portion again, and found a loss which, even after making the Somerset House correction, amounted to -2.05 per cent., while Mr. Adams' portion had undergone comparatively little change. The President's portion was transferred to another bottle during the fortnight, and when analysed the second time had a pleasant beer-like odour, having evidently undergone alcoholic fermentation. This fact would account for the great loss in the solids, and if a fall of 2.05 per cent. can occur in a fortnight, why not a loss of 4.07 per cent. in a month, as in Mr. Estcourt's case? \* Where results in the table were bracketed together, they were obtained by analysis of the same milk at different ages, except in the case of Dr. Vieth's samples, where

\* Since the meeting the sample in question has been again analysed, and at an age of 33 days has sustained a loss of 5.58 per cent. of total solids, which leaves a Somerset House error of -5.03!

the difference in the rate of decomposition, which was unusually rapid, was apparently due simply to the difference in temperature, one portion of the milk being kept at 10-15° C. and the other at 19-21° C. In the case of Mr. Adams' samples, the solids in the fresh milk were determined in duplicate. After 34 days the samples were sent to Somerset House, where the referees kept them another three weeks before reporting. Hence the age given (47 days) was the mean between 34 and 60, at which latter age the certificates were issued by Dr. Bell and his colleagues. In two cases the Somerset House error was more than twice as great as in two others, but in all cases the error was too large to be tolerated.

It was quite possible that in some cases the errors in the analyses of Dr. Bell were increased by the difficulty of taking a fair sample of the curdled milk, and the same objection applied to all such analyses of decomposed milk, as usually made. He was, therefore, instituting a series of experiments in the following manner:—5 c.c. quantities of milk of known specific gravity were placed in a series of test tubes, which were then duly closed and preserved. One of these was every week opened, the contents completely removed to a dish, evaporated, and the residue weighed. A series of twelve tubes would enable him to examine the rate of decomposition of a sample during three months, with a minimum of labour.

Doubtless one of the great difficulties in interpreting the results yielded by decomposed milk was the various kinds of fermentation which could occur, and the impossibility of predicting which of these would occur or predominate. As a rule, the lactic fermentation proceeded most actively, but it was almost always complicated with alcoholic and butyric fermentations. These latter formed volatile products, and hence a great diminution of the total solids, but such facts did not prevent the Somerset House chemists from applying their constant allowance to a milk which they analysed when eighty days old, and which was stated in their certificate to be in "an advanced state of decomposition, having gone beyond the lactic fermentation" (ANALYST, i., 75). Notwithstanding this, however, by a happy coincidence the solids thus corrected differed only by 0.01 per cent. from the determination made by Mr. Jones on the fresh milk—an agreement which, under the circumstances, speaks as eloquently against the value of the allowance as do the enormous discrepancies in other cases.

Mr. ADAMS said one of the greatest difficulties in dealing with sour milk was the sampling. He would like to ask Mr. Stokes whether he accounted for the monstrous difference between 2.38 per cent. of fat and 1.74 per cent. in the sour milk by that difficulty. He himself had had much trouble in dealing with sour samples; do what he might, he found that the fat would adhere to the vessel or else distribute itself unequally—in lumps—through the sample.

Referring to the preservation by ammonia which he formerly brought before the Society, he had had some results which led him to infer that the ammonia caused fat to separate from the solids not fat. After further experiments he had found that the temperature of an ammoniated milk should be raised to about 100° F., and the sample then vigorously shaken, when the fat should become equally distributed, so that he was now more in favour of preserving samples by ammonia than he had been some time since.

Referring to a case mentioned by the President, he had analysed the same milk on the same day, and found the loss to be only .69. He thought that was an important point, as it showed that the same milk kept in two laboratories might change in a perfectly different manner.

His original analysis was	11.82	;	Mr. Allen's,	11.75
After 15 days	11.13		"	11.51
Loss	.69		"	.24



He had some other samples of exactly the same age, and kept under the same conditions, the loss in these being '45 and '96.

Dr. VIETH said the question of allowance ought to be disposed of without much discussion. There was no possibility of calculating the decomposition which might occur in a sample of milk. The question had, however, been raised in a quarter which public analysts could not ignore, and even if they could kill the question, ways and means would be found to bring it back again. As Dr. Bell stated in his book that he had based his allowance on practical experiments, he thought it was very well that Mr. Stokes' experiments should be brought before the Society, to show the fallacy of Dr. Bell's assumption. He made some experiments four or five years ago, but thought they would scarcely bear on the question, as he worked them in a different way. He divided a sample of milk on platinum capsules, put 5 c.c. on 10 capsules, kept 5 of them in the laboratory, and five others in an ice safe, and weighed them at intervals, and found a reduction took place. However, he did not think those experiments bore on the keeping of milks in the hands of analysts. He did not believe in the President's suggestion of taking the total solids only, because he thought it was impossible to redistribute the fat after it had been standing for any length of time; the only way would be, to warm the milk, which again would curdle it and make it impossible to redistribute the casein. According to the advertisements at shows of glaciated and other similar things, he must take it that they are very largely used, and in London it was rather difficult to buy a milk from a dealer which did not contain any preservative, and, of course, those milks did not decompose so quickly.

Decomposition was due to the milk-sugar, which, very curiously to note, might decompose in two different ways, either by splitting up into lactic acid, or into alcohol.

Dr. VIETH gave some instances of each.

Mr. LLOYD, Chemist to the Dairy Farmers' Association, said he was glad to be able to say a few words on the paper, which was not only of much interest to him, but was of double importance to him because he had a large number of samples sent him from farmers which are turned, and they wanted to know if they were genuine, and, on the other hand, he had a number of samples which were still sweet when reported upon. He thought Mr. Stokes had opened up a subject which could not be allowed to rest until something more definite had been obtained. He would like to know whether the samples had been preserved in absolutely tight bottles, because if not there might be entirely different changes taking place and resulting in the enormous loss and forming butyric acid, but if the milk is preserved in a tight bottle and butyric acid is formed, then the bottle must be broken. Dr. Vieth had said that alcoholic fermentation did not take place in milk except under certain circumstances. Subsequently the lactic acid might be split up into butyric acid, and then the bottle would burst. If the bottle were not tight then there would be such losses as Mr. Stokes gave in No. 19.

They had to remember that the samples sent to Somerset House ought to be in tight bottles, and therefore they must put aside this butyric acid and the possibility of its having taken place.

As regards artificial heating, he had tried all temperatures up to boiling point, and he had found that there was a material difference in the change which took place. If they could only settle the question of preservation in a manner which could be carried out by inspectors, it would put aside this question of allowance. One result he had obtained might throw some light on the matter. In all his experiments where the bottles had corks his results were all right, but when they were not well corked he obtained very different results.

One other important consideration he would mention, viz., as to the opening of the

bottles once or twice. They all knew how important the opening of bottles was, causing changes which would never have taken place had the bottle not been opened.

It had been laid down experimentally that the milk-sugar split up into two molecules of lactic acid, consequently, there should theoretically be no loss. After a large number of experiments he had found in only two cases the milk had not undergone any material loss although lactic acid had been formed; the loss in total solids was  $\cdot 02$  and  $\cdot 07$ .

Other samples he had kept

One for 7 days, on which the loss was  $\cdot 38$ ; lactic acid,  $1\cdot 11$ .

Another for 14 " " "  $\cdot 42$  " "  $1\cdot 19$ .

He had taken a large number of samples from 14 days to 40 weeks, and he found, whatever the loss might be (and he had obtained losses from  $\cdot 57$  up to  $1\cdot 20$  per cent. on the total solids), the lactic acid was  $1\cdot 17$  and  $2\cdot 14$ .

Taking these figures, they represented approximately that the loss is almost exactly half of the total amount of lactic acid present in the milk. If fermentation has taken place, that did not hold good for a moment. He had not been able to explain the chemical changes which produced this loss, but if they considered that the ferment was living upon the nitrogenous organic matter in the milk, they should assume that the amount of nitrogen consumed should be in proportion, and consequently if nitrogenous gases were formed during that fermentation the loss would be in proportion to the amount of lactic acid.

He trusted the idea might lead other members who are interested in the subject to make experiments in a similar way.

Mr. CASSAL said he should like to point out that, as a practical Society, they had almost written enough, and they ought now to take some practical action similar to that the President was taking. The results obtained by Mr. Stokes were, no doubt, valuable, but we ought to demonstrate to the public that the Somerset House analysts had assumed a position which they had no right to assume.

Mr. ALLEN, in concluding the discussion, said that one of the samples he referred to smelt like butter, which would probably account for the low results. Dr. Bell made two extraordinary admissions in his letter, and if they could only induce him to write a few more letters they would have nothing to answer.

#### THE SUBSTITUTION OF ASBESTOS CLOTH FOR BLOTTING PAPER IN MR. ADAMS' MODIFICATION OF MR. ABRAHAM'S PROCESS OF MILK ANALYSIS.

By DR. W. JOHNSTONE.

(*Read at the Meeting, November 9th, 1887.*)

MR. PRESIDENT AND GENTLEMEN,—In appearing before you this evening I must at once apologise for having practically, so to speak, no paper to bring before you, as the subject of milk analysis has been so often before the Society. However, I am sure you will accept my apology, when I tell you that the information I intended to communicate to you this evening is already in your hands, viz., "The Substitution of Asbestos Cloth for Blotting paper in Mr. Adams' modification of Mr. Abrahams' process of Milk Analysis."

The following figures represent the results obtained from samples of milk analysed in my laboratory in the ordinary course, and therefore not selected for the occasion.



Specific Gravity.	Total Solids.		Fat Found.		Fat Calculated.
1029.4	11.810	11.821	3.244	3.143	3.465
1030.3	12.417	12.411	3.433	3.402	3.780
1031.2	12.350	12.324	3.250	3.270	3.508
1031.7	12.870	12.820	3.598	3.632	3.810
1029.4	11.97	11.94	3.260	3.248	3.408
1033.8	11.60	11.65	1.630	1.627	
1036.2	10.81	10.82	0.740	0.752	

The specific gravity was carefully taken at 60° F. and the total solids obtained in duplicate from about 5 grms. of milk.

The fat was extracted from the residue of about 10 grms. of milk by ether in an ordinary Soxhlet's tube, the milk taken having been absorbed by a coil of asbestos cloth 12 inches long by 2½ wide and thoroughly dried previous to commencing the extraction. (Here is one of the coils.) The advantage to be obtained in the use of asbestos cloth, is that, after having been thoroughly ignited, the operator has a material of great porosity, which is absolutely insoluble in ether, and therefore can yield no ethereal extract whatever. It is therefore a very suitable material to use in extracting the fat from a milk residue, as the size of coil mentioned easily and rapidly absorbs 10 grms. of milk, and when dry the fat is readily extracted from the thin film of the milk residue distributed on the asbestos. Another advantage is that the coils can be used an indefinite number of times; all that is required to be done, is simply to burn off the milk residue, allow them to cool, when they are again ready for use.

*Conclusion of the Society's Proceedings.*

## ON REICHERT-MEISSEL'S METHOD OF BUTTER ANALYSIS, AND ITS APPLICATION FOR THE EXAMINATION OF BUTTER AND BUTTER SUBSTITUTES.

BY DR. RUDOLF WOLLNY.

*(Continued from page 210.)*

The following arrangement was devised to take the requisite volume of alkali from the bottle without risk of absorption of carbonic acid. One aperture of a Woulff's bottle carries through a cork a glass tube, 2 c.m. in width and 5 c.m. in length, filled with soda-lime. Through the other aperture of the bottle (capacity about 1 litre) goes a glass tube through a cork to the bottom of the bottle. The upper end of this tube is joined by means of a narrow india-rubber tube, which carries a pinchcock, with the lower end of an ordinary burette, into which, near its lower end, a side tube has been blown, carrying a pinch-cock, india-rubber tube, and a glass nozzle. The upper end of the burette carries a soda-lime tube. The whole arrangement is firmly joined together by means of a board. The burette is filled by sucking at the upper soda-lime tube, whilst the pinchcock at the pipe which communicates with the bottle is opened.

Soda solution, which is made and kept as described, furnishes from 3 c.c., no more volatile acid than will neutralise .2 to .3 c.c. deci-normal solution.

It was next necessary to prevent absorption of carbonic acid during saponification. This could have been readily managed by saponifying in a corked flask carrying a suffi-

ciently long glass tube, the upper end of which is bent downward; through this, the alcohol vapours could escape, whilst carbonic acid could not enter. The strong odour of butyric ether which is always observed when butter is saponified, however, proves that the Reichert-Meissl method has a second source of error, butyric acid being lost when alcoholic potash is used.

(*Note by Translator.*—This has long been pointed out by Hohner, Allen, Wanklyn, and others.)

As butyric ether is decomposed by heating with alcoholic potash, it seemed advisable to heat the mixture for some time under a reflux condenser. The following experiments will show that in this manner the ether is completely decomposed, and the butyric acid retained.

Experiment 102.—5 grms. butter were saponified with 3 c.c. NaHO and 6 c.c. alcohol in a flask connected with an ordinary condenser, the alcohol being distilled into a second flask. The soap dissolved in 100 c.c. water, and distilled with 40 c.c.  $H_2SO_4$  (1 : 10), required for 110 c.c. distillate 27.86 c.c. deci-normal solution.

Experiment 103.—The distillate obtained in 102 smelling strongly of butyric ether, was heated with 3 c.c. NaHO under reflux condenser for half an hour. The alcohol was then distilled off, the residue diluted with water, and distilled with  $H_2SO_4$  as above. 100 c.c. distillate required .69 c.c. deci solution.

Experiment 104.—5 grms. of the same butter were heated with NaHO, and alcohol in the same proportions as in 102 for half an hour under reflux condenser. 110 c.c. of the soap distillate required 28.52 c.c. deci-solution, that is, the sum of the quantities used in 102 and 103.

In other similar experiments, a quantity of butyric ether was obtained which neutralised respectively .2, .2, .4, .9, 1.5, and 1.7 c.c. deci-solution, the amount of ether being the larger the greater the volume of NaHO and alcohol.

The two chief sources of error of the Reichert-Meissl method are therefore a loss of butyric ether, and a gain by absorption of carbonic acid, and from what has been said, it follows that the saponification must be carried out under a reflux condenser, that the alcohol must be distilled off from the closed flask, and that the soap must be dissolved in water, also in a closed flask.

I have devised the following arrangement in order to combine these various operations in a convenient manner. A condenser slanting upward at an angle of  $45^\circ$  is fixed near the water-bath upon which the saponification is to take place. The flask is connected with the condenser by means of a T-piece and india-rubber tubes, so that the leg of the T-piece can be directed upward or downward as desired. During saponification, which should take half an hour on the boiling water-bath, the leg of the T-piece is directed upward, being closed with a short piece of india-rubber and glass-rod. The alcohol in this manner runs back into the flask. After that time the T-piece is turned downward and opened: the alcohol can thus be collected in a flask standing beneath. After twenty minutes, when distillation is complete, the T-piece is again turned upwards, and through it 100 c.c. of boiling water are run into the flask by means of a pipette being tightly joined to the short piece of india-rubber. The T-piece is closed again until the soap is completely dissolved in the water, solution being assisted by gently



shaking the flask. The flask is then taken off the water-bath, its contents cooled under the water-tap to 50–60° (not lower), 40 c.c.  $\text{H}_2\text{SO}_4$ , and a few pieces of pumice are added, 110 c.c. distilled off, filtered, and 100 c.c. titrated with deci-normal baryta water, phenol-phthaleine being the indicator. I prefer baryta to alkali solution, because with it the change of colour is somewhat sharper, although the colouration produced is rather pink than purple. Baryta has also the advantage that it at once indicates if accidentally some sulphuric acid has been mechanically carried over into the distillate. The baryta solution should be kept in a Woulff's bottle precisely as the 50 per cent. soda solution previously referred to. A few blank experiments should be made, and the small quantity of volatile acid obtained subtracted from the figures yielded by fats.

The nozzle of the soda burette must, previous to each experiment, be wiped clean from any carbonate, and the first few drops which issue must be rejected. The alcohol must not be blown into the flask by means of the pipette; no respired air must be allowed to enter; all vessels used must be absolutely clean and neutral.

(To be continued.)

#### ESTIMATION OF POTASSIC BITARTRATE IN WINE-YEAST AND CRUDE CREAM OF TARTAR.

By ARTHUR BOENTRÄGER.\*

Two processes are most commonly used: The direct titration with standard soda and Röhrig's process *à la Casserole*.

##### THE DIRECT ESTIMATION.

This process has been often objected to because the tartar often contains other bodies of an acid nature, which are even sometimes fraudulently added, to deceive the analyst. Warrington, who has thoroughly investigated the process, often obtained results in excess of what was expected from the amount of potash.

##### THE METHOD *à la Casserole*.

I was not able to get the original paper, and therefore had to be content with consulting some abstracts, which did not, however, well agree with one another. According to one abstract 50 grammes of the coarsely powdered tartar must be boiled for ten minutes with one litre of water. The fluid must then be at once poured off, and after standing for twelve hours at normal temperature, the crystals must be washed with cold water, then dried and weighed.

According to another abstract, 50 grammes of the crude tartar are boiled for ten minutes with 1250 c.c. of water, the fluid allowed to stand for two minutes, and then carefully decanted from any insoluble matter. After standing for six hours the crystals are collected, and then three times washed with cold water, using altogether one litre of washwater. They are then dried and weighed. In analysing yeast, the hot liquid should be poured through a fine sieve. As by this process the crystallisable matter is simply weighed and considered to be pure potassic bitartrate, it must in many cases only be a crude approximation. An allowance is made for solubility of the bitartrate

\* *Repert. Anal. Chemie*, 37, 87.

in the mother liquor, but the amount varies considerably with the temperature. To practically ascertain the amount dissolved, the easiest plan is to make an experiment side by side with pure cream of tartar. Boil a sufficiency of pure potassic bitartrate with about 100 parts of water for ten minutes, let crystallise, and by means of standard soda estimate the amount of the salt retained in one litre. We then know how much to allow in our analysis of the crude sample.

The following example will show the necessity of making a proper allowance. Suppose the crystallisation took place at a temperature of 28.29 deg. C., the amount of potassic bitartrate contained in the 1000 c.c. mother liquor will be no less than 8.54 grammes; whilst if the temperature had gone down to 11.5-13.5 degs. C., only 4.1 grammes will be retained. It therefore is not fair to simply make an allowance of 10 per cent., as some analysts are in the habit of doing.

An improvement in the process consists in washing the crystals with a cold saturated solution of pure cream of tartar instead of plain water. But a great source of error is caused by calcic tartrate. According to various authorities, neutral calcic tartrate is soluble in 300-400 parts of boiling water; but it is much more freely soluble in solution of cream of tartar. The greater part of the calcic tartrate must, therefore, crystallise out with the cream of tartar and make the *rendement* too high, unless the crystals are subjected to further analysis. I now made the following experiments:—

The potassic bitartrate I used consisted of a once recrystallised cream of tartar, which contained but unweighable traces of lime, sulphates, and chlorides. To make perfectly sure 2.5 grammes were titrated with normal soda, and took 13.5 c.c., whilst the ash of another 2.5 grammes, when titrated with semi-normal sulphuric acid, also took 13.5 c.c., which proved the perfect purity of the salt.

I now prepared pure calcic tartrate as follows:—

A clear solution of pure Rochelle salts was precipitated with a perfectly pure and carefully neutralised solution of calcic chloride. The precipitate which soon became crystalline was first washed with water until free from chlorine, then with alcohol, finally with ether, and dried at 50-60 deg. C. To test the purity of the salt, two grammes were ignited (once with addition of a little pure sugar) before the blowpipe, and I obtained 15.56 and 15.55 per cent. calcium. The percentage calculated from the formula  $\text{CaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$  is 15.38.

By my oxalic acid process (*Zeitschr. f. Anal. Chemie*, 1887) I got 57.59 per cent. tartaric acid, theory requiring 57.69 per cent. 50 grammes of the pure cream of tartar were boiled, with and without addition of 5 grammes of the pure calcic tartrate, in a porcelain dish with 1100 c.c. hot water, for ten minutes. After standing at rest for two minutes, the liquid was decanted, and the residue was three times boiled with 50 c.c. of water, which liquids were added to the chief mother liquor. The liquid was allowed to stand over night, was then filtered off (filter and basin were weighed together), and the deposit dried, and weighed. The crystals in the experiment with the mixed tartrates were slightly washed with some of the mother liquor of the other. The amount of potassic bitartrate in the mother liquor was now estimated with normal soda.

The crystals obtained from the mixed tartrates were analysed as follows:—



The acid tartrate was estimated by dissolving 2.5 grammes and titrating with normal soda. To estimate the calcium, 5 grammes were incinerated, and the char exhausted with water. The residue was then burnt to white ash and dissolved in hydrochloric acid. The acid was mixed with the watery solution and, after boiling the calcium, was estimated by precipitation with ammonia and ammonic oxalate, and finally weighed as oxide.

In every experiment with pure potassic bitartrate the sum of the weight of the crystals plus the allowance came very close to the quantity originally taken, but when 5 grammes of calcic tartrate had been added the results were far too high. Accurate results were of course obtained by titrating the actual amount of potassic bitartrate in the crystals with normal soda. If to the quantity so obtained is added the weight of the calcic tartrate calculated from the calcium, the weight exceeds the original weight taken. This is caused by the drying. Calcic tartrate readily parts with a considerable amount of its water of crystallisation at a temperature of 100 deg. C.

TABLE I.

Calcic tartrate used.	Weight of Crystals.	Pure bitartrate therein contained.	Calcium found.	Crystallised Calcic tartrate.	Allowance bitartrate.
None.	45.3	45.3			4.71
"	44.3	44.3			5.61
5	48.5	44.2	.6997	4.5481	5.66
None.	44.5	44.5			5.33
5	49.2	45.4	.6612	4.2978	4.78
None.	44.4	44.4			5.31
"	44.4	44.4			5.43
"	45.3	45.3			4.69
5	49.1	45.4	.6039	3.926	4.80
None.	45.0	48.0			5.01
5	48.7	44.5	.6925	4.5013	5.44

From this table it is plain the results obtained by the *casacrole* are much too high. It has been argued this does not matter much, as the calcic tartrate is also a valuable product; but still, it contains only 57.69 per cent. of tartaric acid, whilst the bitartrate contains 79.79 per cent., and for many purposes we want to know the percentage of real potassic bitartrate.

I now felt anxious to know how a mixture of cream of tartar and gypsum would behave in the crystallisation process. I therefore made a similar experiment as before, using 50 grammes of the pure bitartrate, and also 50 grammes of the same, mixed with 6.5 grammes of gypsum. In the blank experiment I obtained 42.85 grammes of crystals, and the mother liquor had retained 6.33 grammes. In the mixture I got 41.80 grammes of crystals, which were analysed as follows:—

2.5 grammes were dissolved in hot water, and titrated with normal soda. They required 12 c.c. equivalent to 2.2541 potassic bitartrate. The total amount was, therefore, 37.69 grammes = 30.08 grammes tartaric acid.

2.5 grammes were boiled under upright condenser with 30 c.c. water and 10 c.c. of a 20 per cent. solution of potassic carbonate for one hour. After cooling, the liquid was made up to 100.5 c.c. 50 c.c. of this were evaporated to 10 c.c., mixed first with 2 c.c. of glacial acetic acid, and after five minutes, with 100 c.c. of 98 per cent. alcohol. After standing over night, the deposit was filtered off, washed with alcohol, then redissolved in hot water, and titrated with normal soda. Used 6.48 c.c. = .9705 gramme tartaric acid in 1.25 of the salt, a total 32.45 grammes tartaric acid.

5 grammes of the salt were carefully charred, the mass extracted with water, and the residue burnt to ash. The ash was treated with hydrochloric acid, which was now added to the watery extract. After filtering, the sulphuric acid was, as usual, precipitated with baric chloride. I obtained .0200 gramme  $\text{BaSO}_4$  = .0148  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  or .1237 in the crystals.

As the crystals contained 32.45 grammes of tartaric acid, of which 30.08 grammes existed as potassic bitartrate, 2.37 grammes must be present as lime compound. The composition of the crystals was, therefore—

37.69	potassic bitartrate
4.11	calcic tartrate crystallised
.12	calcic sulphate crystallised
<hr/>	
41.92	

The residue insoluble in boiling water was nicely crystalline, and looked like pure calcic tartrate, which it nearly proved to be on analysis. The mother liquor had a much stronger acid reaction than the one got in the experiment without the gypsum. The total acidity was equal to 8.38 grammes tartaric acid, showing the presence of free acid. This was tartaric, and not sulphuric, acid; then it gave no blue colour with dimethylaniline violet. The reaction between the cream of tartar and the gypsum seems to be—



The presence of plaster, therefore, considerably affects the *rendement* of the crystals, and renders the process inaccurate.

#### INDIRECT PROCESSES.

Several processes have come to my notice, which were originally recommended for tartars only, but have also been applied to wine yeast. Ten grms. of the substance are incinerated, and the char extracted with water. The solution is evaporated to dryness, and the salts (potassic carbonate) calculated to potassic bitartrate; or, instead of merely weighing, the alkalinity of the ash may be taken with normal acid and then calculated to cream of tartar; or the potash may be determined with platinic chloride. None of these methods will be always successful when the yeast is in a decomposed state, or when the tartar contains much gypsum, or when fraudulent additions have been made. The potassic carbonate formed during the incineration of the sample will act on the gypsum and form calcic carbonate and potassic sulphate. The result obtained by the titration with acid must, therefore, be too low, but this may be remedied by estimating the *sulphate* of potash and calculating this also to the bitartrate, but the process then becomes rather tedious. According to Warington a certain portion of the sulphate is, during the



charring, reduced to sulphide. This may be remedied by first treating the char with a little hydrogen peroxide, but this is no longer practicable when the tartars or yeast contain free sulphur, which is sometimes sprinkled on the grapes. As, however, calcic sulphide, on heating with potassic carbonate, yields calcic carbonate and potassic sulphide (which is as alkaline as the carbonate) I cannot see how the reduction of the sulphate can influence the result of the titration. On applying the titration process to materials in a state of decomposition, it is as well to make an estimation of the carbonic acid in the original sample, but it is difficult to decide how this acid is distributed between the potash and the lime. Oliveri estimates the cream of tartar in yeasts as follows:—10 grms. of the sample are digested with 40 c.c. of water and 5 c.c. fuming hydrochloric acid for 24 hours, then filtered off, and titrated with standard baric chloride. The sulphuric acid thus found is calculated to calcic sulphate. The liquid is now filtered from the baric sulphate and divided into two equal parts. Part *a* is made alkaline with ammonia, and precipitated with ammonic oxalate, to get the lime; part *b* is first nearly neutralised with caustic soda, then mixed with slight excess of ammonia, and then precipitated with calcic chloride. The calcic tartrate is not weighed as such, but ignited, and then converted into sulphate. Any lime not existing as sulphate is supposed to exist as tartrate, and the balance of tartaric acid is calculated to potassic bitartrate. The process is fairly good, but it is perfectly useless if salts of sorrel, Epsom salts, potassic sulphate, or calcic carbonate have been added.

## DIRECT PROCESSES OF RECENT DATE.

Kämmer neutralises 2 grms. of yeast, or tartar, with caustic soda. To prevent the tartar dissolving any calcic carbonate, the neutralisation must be done in the cold. The whole is now made up to 101 c.c. (1 c.c. being occupied by the insoluble matter) and filtered. In 50 c.c. the total tartaric acid is now estimated as follows:—The fluid is evaporated to a low bulk, and then mixed with acetic acid and alcohol. The precipitate is washed with a 5 per cent. solution of potassic chloride saturated with pure cream of tartar. After washing the precipitate is dissolved in hot water and titrated with semi-normal soda. About this process I want to make the following remarks: The neutralisation of the acid tartrate in the cold is a very slow operation. It took me half an hour to dissolve 2 grms. of finely powdered tartrate in 30 c.c. of water by means of normal alkali. The choice of potassic chloride as washing fluid seems to me to be an unfortunate one, and the washing may just as well be done with alcohol. Then, again, the cold neutralisation will not altogether prevent the acid tartrate from acting on any calcic carbonate which may be present.

The makers of cream of tartar, however, only care to know how much pure acid tartrate may be obtained by re-crystallisation of the crude product, and cannot prevent the acid tartrate acting on any calcic carbonate. To them the process must be in many cases useless. As calcic tartrate is soluble in solution of neutral alkaline tartrates I thought it advisable to ascertain whether in Kämmer's process any calcic tartrate passes in solution and may interfere with the result. I therefore neutralised a very finely powdered mixture of 2 grms. of chemically pure cream of tartar and half a gm. of

crystallised calcic tartrate suspended in 30 c.c. of water with normal potash. This took half an hour in the cold. I now made up to 100.5 c.c., filtered off and evaporated 50 c.c. of the filtrate down to 10 c.c.; 2 c.c. of glacial acetic acid were now added, and after five minutes vigorous stirring, 100 c.c. of 98 per cent. alcohol. After standing over night the precipitate was filtered off, washed with the said alcohol, dissolved in boiling water, and at once titrated with normal soda. I used 5.33 c.c. instead of 5.32 = 100.13 per cent. acid tartrate. The presence of even 20 per cent. crystallised calcic tartrate does not therefore interfere with the estimation, notwithstanding a little calcic tartrate passes in solution. I now tried the process with a mixture of cream of tartar and gypsum. Two grms. of pure bitartrate and .3 gm. of gypsum mixed with 30 c.c. of water were neutralised in the cold with normal soda, which operation took about 40 minutes. The liquid was made up to 100.5 c.c., and filtered off, when 50 c.c. of the filtrate was treated as described. Used 4.67-4.69 c.c. normal soda = 87.98 per cent. potassic bitartrate. The presence of 13 per cent. of gypsum (yeasts often contain as much), therefore, seriously affects the accuracy of the process.

#### F. KLEIN'S PROCESS.

This process, which is a great improvement on the others, is based on the slight solubility of cream of tartar in solution of potassic chloride. It has the advantage of being the best process from a manufacturer's point of view.

1.8-2.2 grms. of the sample are boiled 5 times with distilled water, and the residue washed on a filter with boiling water, until every trace of acidity is removed. The filtrate is evaporated down to 40 c.c., when 5 grms. of potassic chloride are introduced. After vigorous stirring for 15 minutes, the precipitate is filtered off, washed with a 10 per cent. solution of potassic chloride (saturated with cream of tartar) and then titrated with normal soda.

*(To be continued.)*

#### BOOKS, &c., RECEIVED.

AMERICAN Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Druggist's Circular; Hospital Gazette; The Illustrated Sidney News; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Scientific American; Society of Arts Journal.

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ANALYSIS OF FOOD AND DRUGS, AND OF GENERAL  
ANALYTICAL AND MICROSCOPICAL RESEARCH.

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EDITED BY

JOHN MUTER, M.A., Ph.D., F.R.S.E., F.I.C., F.C.S., ETC.,

*Late President of the Society of Public Analysts; Author of "A Short Manual of Analytical Chemistry,"  
and of Text-Books on Pharmaceutical Chemistry and Materia Medica: Public Analyst  
for Lambeth, Wandsworth, Southwark, Rotherhithe, Newington,  
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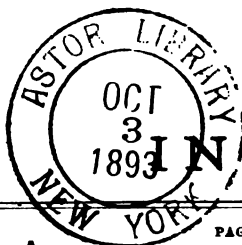
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# THE ANALYST.

JANUARY, 1888.

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## PROCEEDINGS OF SOCIETY OF PUBLIC ANALYSTS.

Ordinary meeting of this Society was held on the 14th ult. at Burlington House, the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened it was reported that the following gentlemen had been elected :—As member, W. Hogben, analytical chemist. As associate, A. W. Trench, Assistant to Mr. O. C. Hagemann.

The following gentlemen were proposed for election :—As members, John Heron, analytical chemist ; W. E. Matthews, analytical chemist, of Melbourne ; Laurence Briant, brewer's analyst, Holborn Viaduct.

Mr. Fox was appointed auditor for the ensuing year, and in case Mr. Johnstone should be too unwell to act, it was decided to ask Mr. Harland to assist Mr. Johnstone.

Mr. Adams proposed, and Mr. Hehner seconded, a notice of motion, to be on the agenda for next meeting, that " Mr. W. F. Lowe, of 13, Cheapside, be appointed Solicitor to the Society, and that whenever legal business is likely to come before the Council he be invited to attend."

The following papers were read and discussed :—

" Note on the Estimation of Peroxide of Hydrogen." By C. T. Kingzett

" The Relation between Specific Gravity, Fat and Solids not Fat, upon the Basis of the Society's Process for Milk Analysis." By Otto Hehner and Henry D. Richmond.

At the instance of Dr. Muter (who was absent through illness), Mr. Hehner referred to the new Margarine Act, which comes into force on the 1st of January, and to the question of public analysts' certificates under it.

1 Paper.	2 Smooth; Shaft full.	3 Folded larger than 90°.	4 New Funnel.	5 Old Fluted Funnel.	6 Pleated Paper.	7 Richmond's Pleat.
Best Swedish, Thin ....	682	592	95	854	56	70
	200	548	95	1290	70	73
	480	138	97	600	61	69
	218	85	74	115	84	60
Average	395	341	90	715	68	68
English, Thin	328	137	51	152	52	75
	306	135	60	90	45	83
	156	79	49	217	80	91
	181	287	65	152	48	55
Average	285	160	56	153	61	76
Schleicherand	563	840	160	338	98	120
Schüll(590),	450	160	145	543	100	200
washed with	545	180	192	582	90	155
H.Fl. ....	750	250	162	508	188	150
Average	577	357	165	492	119	156
Schleicherand Schüll(597), very Thick	1070	415	262	295	192	155
	700	368	350	288	210	172
	612	1125	332	420	180	175
	1160	280	322	488	202	160
Average	886	547	317	373	198	150
Schleicherand Schüll(597), Thick ....	548	517	188	132	85	92
	412	182	212	455	88	100
	388	252	168	188	135	145
	430	252	160	507	80	92
Average	445	301	182	321	97	107

It is seen that, with all classes of filter-paper examined, the worst plan which can be adopted is to fit a filter closely into the funnel. The paper does not get a chance to work, and the loss of time is very great, the greater the thinner the paper. When the filter is folded so that it lies loosely in the funnel, excellent results are frequently obtained, but these are somewhat erratic, especially with thin papers, because the paper is apt to slip closely into the funnel when fully weighted with water. In all cases the velocity in Class 3 was the greater, the greater the angle at which the fold was made.

The new ridged funnel gave the best and most concordant results. It disposes of any chances due to imperfect folding or fitting of the paper; it allows more freely than any of the other methods the passage of the fluid into the stem, and this far more certainly than the old fluted funnel, which, indeed, with thin paper gives very bad results.

Paper folded in pleats in the usual manner filters, especially in the case of very thick paper, faster than smooth filters are capable of doing, but the long, uneven



edge renders washing so difficult that they cannot well be used for exact quantitative work.

The folding used in the tests stated in Column 7 is intended to obviate this difficulty as far as possible, and to provide a pleated filter with a smooth edge. It will be seen that its filtration velocity equals that of ordinary pleated paper.

The funnels with deep ridges, described in this paper, have been made by, and may be obtained from Messrs. Townson and Mercer, 87, Bishopsgate Street, at a price differing but very little from that of ordinary smooth funnels.

### ALUMINA AS A NATURAL CONSTITUENT OF WHEAT FLOUR.

By W. C. YOUNG, F.C.S.

*(Read at Meeting, November, 1887.)*

IN a paper read before the Society last June, and published in the August number of the ANALYST, I gave an account of some experiments with the logwood test, the results of which seemed to indicate that alumina was a natural constituent of wheat flour, and, further, that it was confined to the gluten, the starch being quite free from it. In the discussion which followed, our President pointed out that it was generally supposed that alumina was not a natural constituent of wheat flour, and that when found it was ascribed to accidental impurities or purposely added alum. I find also that in the discussion of a paper by Wanklyn at the first meeting of this Society, Mr. Allen similarly expressed himself, but Dr. Dupré mentioned that in conjunction with Dr. Odling he had made many analyses of wheat, and had found minute quantities of alumina in every sample.

Recently Yoshida has communicated a paper to the Chemical Society on "Aluminium in the ashes of flowering plants," in which he shows that alumina is a normal constituent of wheat and other cereals.

Soon after the reading of my last paper, I made a quantitative experiment on wheat flour, the result of which not only confirms Yoshida's work, but shows further that the whole of the alumina is contained in the gluten.

The flour used was the best quality Vienna, containing .7 per cent. of ash, and as near as I could ascertain about 8 per cent. of gluten. I obtained from 100 grammes of this flour, by a process I shall presently describe, .0075 gramme of phosphate of alumina.

The gluten was separated by washing in a muslin bag in the usual way, and when dried contained 1.26 per cent. ash; 20 grammes of this dried gluten, finely powdered, was then treated with about 250 c.c. of a mixture of equal volumes of acetic acid and water, and heated in the water bath for about twenty-eight hours. By this time the mass had become quite liquid, the gluten having lost its firmness in the same way that gelatin does under similar circumstances. After standing a short time the liquid was poured off, and the sediment further treated with weak acetic acid twice, and the three portions of liquid evaporated to dryness, the sediment being rejected. In this way I think that any extraneous earthy matter present in the gluten was separated, and, therefore, only the natural alumina retained.

The dried residue was then burnt to a perfect ash, the ash dissolved in dilute

hydrochloric acid and filtered, the insoluble matter being well washed and weighed. The insoluble matter thus obtained weighed only .009 gramme, and of this .0075 was silica.

The insoluble matter was then fused with about twice its bulk of mixed alkaline carbonates, dissolved in dilute hydrochloric acid and filtered. This filtrate was added to the acid solution of the ash, evaporated to dryness, redissolved in a small quantity of dilute hydrochloric acid and filtered. The filtrate was then boiled, and cautiously added to 25 c.c. of a saturated solution of pure caustic soda, also boiling, and the whole kept boiling for a few minutes. It was then filtered, and the precipitate washed, the filtrate made slightly acid with hydrochloric acid, about 5 c.c. of a saturated solution of sodium phosphate added, and finally a slight excess of ammonia. After boiling for about ten minutes, the precipitate of phosphate of alumina was collected and weighed.

I may mention that the process I have just described has been in use for some years now in my laboratory for determining alumina in bread and flour, and is really an improvement on a modification of Normandy's old process which I suggested some years back. The points to be observed as essential to success are, first, the fusion with alkaline carbonates of the ash insoluble in hydrochloric acid, as I have repeatedly found that hydrochloric acid does not dissolve the whole of the alumina in the ash; second, keeping the solutions down to the smallest possible bulk; and third, the employment of a saturated solution of soda.

In this way I obtained .0185 gramme of phosphate of alumina from 20 grammes of gluten. Now as the flour contained 8 per cent. of gluten, and gave originally .0075 per cent. of phosphate of alumina, 20 grammes of gluten would be equivalent to 250 of flour, which would yield .01875 of phosphate of alumina. So that practically I obtained the whole of the alumina of the flour in the gluten. As in the process of washing the starch from the gluten a large proportion of any foreign earthy matter that may have been present must have been separated, and any remaining eliminated by dissolving the gluten in acetic acid, there can be no doubt that the alumina obtained in this experiment was present as a natural constituent of the flour, and I think further that the interesting fact is established that the bulk of it is associated with the gluten.

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#### DOES NEUTRAL OR SUB-ACETATE OF LEAD PRECIPITATE HOP-BITTERS?

By DR. W. JOHNSTONE.

(Read at Meeting, November, 1887.)

GENTLEMEN,

It will be in your recollection that our worthy President read a paper before the Society in May last, entitled "An Improved method of Detecting Quassia and certain other Hop-substitutes in Beer," wherein the following sentence appeared: "Seeing that the bitter principles of hops are entirely precipitated by neutral acetate of lead, the presence of some hop-substitute is absolutely certain if the chloroform or other residue *has a marked bitter taste.*" In the interesting discussion following the reading of that



paper, Mr. Adams said he had no difficulty in distinguishing between a bitter of hops and the substitute used for it, but he did not think there was any possibility of distinguishing between the individual bitters, and one could only positively say there was another bitter present besides that of hops. Dr. Muter, in following Mr. Adams, remarked as follows: "Now came a difficulty which shook his faith as an analyst as regarded hop bitters. He had always believed in the process, from practising upon beers with various added bitters, until some time ago he got a beer which he was privately assured by the maker to have a bitter other than hops. This sample he put through the process, and he got a bitter out of that beer with chloroform, after using lead. He worked on a fairly large quantity, but the process here showed bitters other than hop, although he was assured that the sample represented as pure a beer as could possibly be obtained."

I, Mr. President, have experienced and obtained the same results as Dr. Muter, and have now no hesitation in stating that the use of neutral or sub-acetate of lead in precipitating hop bitters is of no use whatever, and for the following reasons: in that I received a sample of beer from my friend, Mr. Quartermain, who assured me that it was brewed by himself, and from nothing but pure malt and hops. Now this sample of beer when put through the process gave a decided bitter.

The result aroused my suspicions, and I wrote to my friend reporting the result, when he kindly replied, assuring me again that the beer was as he represented, and that there were four different samples of hops used in the brew, three of which he sent me, at the same time stating that he was very sorry he could not send the fourth sample, as they had been entirely used up at that particular brewing, a circumstance I also regretted; however, I submitted the three samples I received to the process and found all of them gave a distinct bitter, so under these circumstances the bitter found in the beer could not be wholly derived from the missing sample, and therefore does not affect the finding of the bitter in this particular sample of beer.

The idea now occurred to me, could the three samples of hops examined have been treated in any way with an infusion of Quassia or other foreign bitter; so to decide this question, if possible, I obtained from Mr. Spriggs, who resides at Maidstone (brother of one of my assistants), a sample of this year's hops, plucked by himself and forwarded direct to me in the condition in which they were picked, and upon arrival they were carefully wind-dried, and then submitted to the process. The results obtained being a decided and distinct bitter.

One fact more, and I am finished. This morning I received a sample of hops from our President, who, in the letter that accompanied the sample, informed me that he could obtain no bitter from them after having submitted them to the process. I, fortunately, have been able also to pass them through the mill, but with quite a different result. Here, gentlemen, is the bitter extracted for you to taste.

I shall not detain you any longer, gentlemen, but conclude by stating that the process described by our President in his paper, read before the Society and published in the June number of THE ANALYST, is quite misleading, and that the use of neutral or sub-acetate of lead, used in the manner as therein described, does not precipitate the natural matter belonging to hops.

*(Conclusion of the Society's Proceedings.)*

# ON REICHERT-MEISSL'S METHOD OF BUTTER ANALYSIS, AND ITS APPLICATION FOR THE EXAMINATION OF BUTTER AND BUTTER SUBSTITUTES.

BY DR. RUDOLF WOLLNY.

(Continued from page 237.)

THE following results were obtained by the method improved as described. The reagents were first tested, and then five blank experiments were made, 3 c.c. NaHO and 6 c.c. alcohol being treated without fat, precisely as the sample to be analysed, namely, they were boiled for 15 minutes under a reflux condenser, the alcohol distilled off in 15 minutes; 100 c.c. of boiled distilled water were added, and the solution kept hot in a water bath for the same time, and ultimately distilled with 40 c.c.  $H_2SO_4$  and two small pieces of pumice; the distillate, measuring 110 c.c., was filtered, and 100 c.c. were titrated. The figure thus obtained, due to the volatile acid contained in the soda solution and to the carbonic acid unavoidably absorbed, was subtracted from the analytical results furnished by the various fats. In the second column of the subjoined table the corrected numbers are found, indicating exactly the *volatile fatty acids*.

ANALYSES MADE BY R. WOLLNY.

C.c. deci-normal alkali

No.	Substance examined.	Consumed	Corrected.	Calculated
105	3 c.c. NaHO	.30	—	—
106	" "	.30	—	—
107	" "	.30	—	—
108	" "	.30	—	—
109	" "	.25	—	—
110	Blank experiment	.35	—	—
111	" "	.35	—	—
112	" "	.30	—	—
113	" "	.30	—	—
114	" "	.25	—	—
115	Beef fat	.33	.03	—
116	"	.33	.03	—
117	Earthnut oil	.33	.03	—
118	" "	.33	.03	—
119	Sesam Oil	.33	.03	—
120	" "	.33	.03	—
121	Cotton Oil	.44	.14	—
122	" "	.39	.09	—
123	Oleomargarine	.39	.09	—
124	"	.39	.09	—
125	Pure Butter	28.82	28.52	—
126	" "	28.93	28.63	—
127	" "	28.71	28.41	—
128	" "	28.93	28.63	—
129	" "	28.88	28.58	—
130	" "	28.82	28.52	—
131	" "	28.82	28.52	—
122	" "	82.77	28.47	—



Substance examined.	C.c. deci-normal alkali.		
	Consumed.	Corrected.	Calculated
Pure Butter	28.82	28.52	—
" "	28.88	28.58	—
" "	20.45	20.15	—
Margarine 1 (2 per cent. Butter)	.88	.58	.62
" " ( " " " )	.88	.58	
" 2 (4 " " )	1.54	1.24	1.20
" " ( " " " )	1.43	1.13	
" 3 (6 " " )	1.98	1.68	1.77
" " ( " " " )	2.04	1.74	
" 4 (8 " " )	2.64	2.34	2.34
" " ( " " " )	2.70	2.40	
" 5 (10 " " )	3.19	2.89	2.92
" " ( " " " )	3.30	3.00	
" 6 (50 " " )	14.63	14.33	14.35
" " ( " " " )	14.74	14.44	
" 7 (85 " " )	24.64	24.34	24.35
" " ( " " " )	24.86	24.56	
" 8 (90 " " )	26.51	25.21	25.79
" " ( " " " )	26.18	25.88	
" 9 (95 " " )	27.61	27.31	27.22

## ANALYSES MADE BY A. SANGER.

3 c.c. NaHO	.17	—	—
Blank Experiment	.22	—	—
" "	.22	—	—
" "	.22	—	—
" "	.22	—	—
" "	.22	—	—
Oleomargarine	.28	.06	—
"	.28	.06	—
"	.28	.06	—
"	.28	.06	—
"	.22	.00	—
Pure Butter	29.25	28.93	—
" "	29.15	28.93	—
" "	29.15	28.93	—
" "	28.82	28.60	—
" "	28.82	28.60	—
" "	28.82	28.60	—
" "	28.99	28.77	—
" "	28.99	28.77	—
Margarine 1 (2 per cent. Butter)	.77	.55	.62
" " ( " " " )	.88	.66	
" 2 (4 " " )	1.54	1.32	1.20
" " ( " " " )	1.49	1.27	
" 3 (6 " " )	2.09	1.87	1.77
" " ( " " " )	2.15	1.93	
" 4 (8 " " )	2.64	2.42	2.34
" " ( " " " )	2.64	2.42	

No.	Substance examined.	C.c. deci-normal alkali.		
		Consumed.	Corrected.	Calculated.
180	Margarine 5 (10 percent. Butter)	3.08	2.86	2.92
181	" " ( " " " )	3.08	2.86	
182	" 6 (50 " " )	14.47	14.25	14.35
183	" " ( " " " )	14.41	14.19	
184	" 7 (85 " " )	24.31	24.09	24.35
185	" " ( " " " )	24.42	24.20	
186	" 8 (90 " " )	25.74	25.52	25.79
187	" " ( " " " )	25.95	25.73	
188	" 9 (95 " " )	27.50	27.28	27.22
189	Cotton Oil	.31	.09	—
190	" "	.35	.13	—

## SUMMARY OF ANALYSES.

Substance examined.	Results by modified method.			Results by Meissl method.		Differences of 4 and 5 compared with 3.	
	Min.	Max.	Mean.	Min.	Max.	Min.	Max.
Beef-fat.	1	2	3	4	5	6	7
Oleomargarine.	.03	.03	.03	—	—	—	—
Earthnut oil.	—	—	—	.26	.92	—	—
Sesam oil.	.03	.03	.03	.19	1.29	.16	1.26
Cotton oil.	.03	.03	.03	.63	1.07	.60	1.04
Butter mixture, 2 per cent.	.09	.14	.11	.74	.74	.63	.63
" 4 "	.55	.66	.59	1.07	1.58	.48	.99
" 6 "	1.13	1.32	1.24	1.84	2.13	.60	.89
" 8 "	1.68	1.93	1.81	2.39	3.60	.58	1.79
" 10 "	2.34	2.42	2.40	2.83	3.34	.43	.94
" 50 "	2.86	3.00	2.90	3.16	3.89	.26	.99
" 85 "	14.19	14.44	14.30	15.04	15.88	.74	1.58
" 90 "	24.09	24.56	24.30	24.94	25.34	.64	1.04
" 95 "	25.52	26.21	25.84	26.04	26.55	.20	.71
Pure butter.	27.28	27.31	27.30	27.76	28.24	.46	.94
	28.41	28.93	28.65	28.93	30.55	.28	1.90

The soda solution used by myself had been filtered through glass-wool, but was slightly turbid from suspended carbonate; that used by Sanger was quite clear, and the latter, therefore, found somewhat less volatile acid in the alkali used than I did.

In experiment 135 the soap solution had been allowed to become cold and had consequently gelatinised. The fatty acids separated from it were therefore solid and fused only towards the end of the distillation. The volatile acids obtained consumed only 20.15 c.c. instead of 28.65 c.c. It is necessary therefore that the soap solution should be decomposed when having a temperature of not less than 50 to 60° C.

The correct figures of column 2 agree very well with the calculated ones. The volatile fatty acids passing into the distillate correspond therefore to the total quantities contained in the soap, and hence it is possible to calculate accurately the percentage of butter contained in mixtures, when the fluctuations of volatile acids in different butters have been ascertained by further experiments. All estimations hitherto made are



fective, for reasons already stated. The same applies to the estimations of volatile acids in the various materials entering into the composition of butter-substitutes. The method of analysis, as modified by me, satisfies all reasonable demands which may be made, and complies with the requirements of Clause 2 of the German Margarine Bill.

The object of the present investigation has so far been directed towards conducting the saponification in a manner free from sources of error; I now proceed to the second part of the research, viz., to ascertain the conditions of *distillation* whereby absolutely uniform results may be obtained. This object has already been attained in materials yielding small amounts of volatile acids; but in the case of butter and mixtures containing a large percentage of it, differences up to .7 c.c. have been observed. It became necessary therefore to study all conditions which might influence the result of the distillation.

In the first place, it appeared of interest to ascertain the total amount of volatile acids contained in butter, respectively how much could be obtained by repeated distillation of the decomposed soap.

Experiments 191-202.—5 grms. butter-fat were saponified and decomposed as usual; after 110 c.c. had been distilled off, 110 c.c. distilled water were added, and the like amount again distilled off; this was repeated twelve times.

The distillates required the following quantities of deci-normal solution:—

1.	28.16	5.	.33	9.	.28
2.	2.20	6.	.33	10.	.28
3.	.77	7.	.33	11.	.22
4.	.50	8.	.33	12.	.22

total of 33.95 c.c., the distillation not being quite completed even then.

Similar series of experiments (203-217) were made with three other samples of butter.

	1.	2.	3.
Distillate 1	26.74	27.99	31.29
" 2	2.97	2.86	3.41
" 3	.88	.77	1.10
" 4	.55	.55	.66
" 5	.44	.44	.44
	<hr/>	<hr/>	<hr/>
	31.58	32.61	36.90

The quantities of volatile acids obtained in the first distillate are therefore fairly proportionate to the total amounts, and no advantage could be derived for analytical purposes by repeated distillations.

The molecular weight of the volatile acids contained in the various fractions of butter 3 were next ascertained by weighing the baryta salts. That of the first and main fraction was found to be 96, whilst the insoluble portion of the fatty acids passing over with the first distillate was 197, and with the further distillates 251. The soluble acids therefore chiefly consist of butyric acid, the insoluble ones correspond with palmitic acid.

Experiment 218. 100 grms. of a 5 per cent. solution of pure butyric acid were distilled and each portion of 10 c.c. of the distillate titrated with normal soda. The various fractions consumed 10.0, 9.4, 8.3, 7.3, 6.2, 5.1, 3.8, and 2.9 c.c., the remainder in the retort, measuring about 20 c.c. using 2.0 c.c. Na.HO.

(To be concluded.)

# ESTIMATION OF POTASSIC BITARTRATE IN WINE-YEAST AND CRUDE CREAM OF TARTAR.

BY ARTHUR BORNTREGER.

(Concluded from page 242.)

A cream of tartar thus treated gave 99.6 instead of 99.87. A mixture of 1.875 grms. of this sample mixed with .625 grm. calcic tartrate and some acid calcic phosphate gave 99.52 per cent. potassic bitartrate. I had the same idea in my mind before Klein published his process, but I always thought it would be necessary to make an allowance for the slight solubility of the cream of tartar in the 10 per cent. solution of potassic chloride. I have since made experiments which confirmed my views. A 10 per cent. solution of potassic chloride retained in 100 c.c. .0376 grm. of bitartrate at a temperature of 11.5-13.5°C. and .0583 grm. at 28-29°C. In Klein's experiments there must, therefore, have been a loss of about 1.66 per cent. of cream of tartar. Further experiments have also convinced me, it makes no difference whether the liquid is filtered off after standing for only half an hour (after ten minutes vigorous stirring), or allowed to stand all night.

## SOLUBILITY OF POTASSIC BITARTRATE IN 10 PER CENT. SOLUTION OF POTASSIC CHLORIDE AT ORDINARY TEMPERATURE.

1.25 grms. bitartrate were dissolved in about 125 c.c. boiling water, and after cooling mixed with 10 grms. of potassic chloride for every 100 c.c., stirred well for 10 minutes, and after standing for half an hour filtered off. The acidity was then taken with  $\frac{N}{10}$  soda in 50 c.c. of the filtrate. In the table the soda is given in c.c. N soda.

TABLE II.

Temperature of fluid—				N. Soda. c.c.	Bitartrate in 100 c.c.
Before adding KCl.	After adding KCl.	After 10 minutes' standing	After standing half-hour.		
26.5°C.	21°C.	23.5°C.	24.5°C.	.13	.0488
..	..	..	..	.13	..
18.5	15	17.5	17.5	.11	.0432
..	..	..	..	.12	..

I further once more determined the amount of bitartrate which separates after standing over night from a solution of 2.5 grms. of cream of tartar is 55 c.c. of water with addition of 5 grms. potassic chloride. The crystals were first washed with a 10 per cent. solution of potassic chloride saturated with cream of tartar. The further washing with 10 per cent. potassic chloride without cream of tartar is superfluous, as will be seen from Table IV.

TABLE III.

Temperature of liquid—			N. Soda. c.c.	Bitartrate found—	
Day.	Midnight.	At time of filtering.		Grms.	Per cent.
23.5°C.	20°C.	19.5°C.	13.06	2.4553	98.21
20.5	19.5	20.5	13.05	2.4534	98.14
22.5	20	21	13.08	2.4590	98.36

The following results were obtained after allowing to stand for only half an hour.



The crystals in the first six experiments were finally washed with a 10 per cent. solution of pure potassic chloride.

TABLE IV.

Temperature of fluid—

Before adding KCl.	After adding KCl.	After stirring.	After filtering.	N. Soda. c.c.	Bitartrate found. Grms.	Per cent.
27.5°C.	23.5°C.	23.5°C.	23.5°C.	13.04	2.4515	98.06
25.5	21.5	22.5	23.5	13.05	2.4534	98.14
17.5	13.5	16.5	17.5	13.05		
..	..	..	..	13.05		
24	20	22	23.5	13.05	2.459	98.36
23.5	15.5	18.5	21.5	13.08		
22	17.5	20.5	21.4	13.04	2.4515	98.06
20.5	16.5	18.5	20	13.05	2.4534	98.14
22.5	18.5	20.5	21.5	13.05		

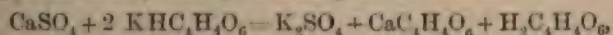
The results of the following table were obtained by treating 2 grammes of cream of tartar with 40 c.c. of boiling water, and after cooling adding the potassic chloride. After 15 minutes stirring and half an hour's standing the crystals were washed with 10 per cent. solution of potassic chloride (saturated with cream of tartar).

TABLE V.

Temperature of fluid—

Before adding KCl.	After adding KCl.	After stirring.	At time of filtering.	N. Soda. c.c.	Bitartrate found— Grms.	Per cent.
23°C.	17.5°C.	21.5°C.	22.5°C.	10.44	1.9627	98.14
..	..	..	..	10.46	1.9655	98.32
16.5	12.5	15.5	17	10.48	1.9702	98.51
..	..	..	..	10.46	1.9665	98.32
..	..	..	..	10.48	1.9702	98.51

I therefore propose to add .0330 gram. of potassic bitartrate to the quantity found by Klein's process before calculating the percentage. As I had found that cream of tartar acts on gypsum most probably according to the formula—



I made experiments to ascertain the influence of gypsum in the analysis of tartar by Klein's process. A mixture of 2 grms. of potassic bitartrate and .3 gram. of gypsum was exactly treated according to Klein's directions. For the first extraction I used 30 c.c. water; for the following I used less. In experiment A, I boiled first for five minutes; in the others, ten minutes, so as to see whether longer boiling makes a difference. The insoluble matter was washed on the filter with boiling water until quite free from sulphates. The filtrates were boiled down to 40 c.c., mixed with 5 grms. of KCl, and treated as usual. Experiment A took 10.27 c.c. N Soda = 1.9308  $\text{KHC}_4\text{H}_4\text{O}_6$  or 96.54 per cent. Experiment B = 10.14 N S or 1.9063 grms. = 95.31 per cent. The presence, therefore, of about 13 per cent. of gypsum makes even Klein's process uncertain.

I now must call attention to another source of error affecting Klein's process and the *casseroles*. Warington has often found in yeasts and tartars a crystallised calcic carbonate, most likely fraudulently added. This is only soluble with the greatest diffi-

culty in cream of tartar, whilst amorphous calcic carbonate is readily attacked. We must, however, not forget that in the process of purifying cream of tartar on the large scale, the time of boiling lasts a good deal longer than in the *casserole* or Klein's method. So to find out the influence of crystallised calcic carbonate (which in samples of tartar may be noticed with a magnifying glass), I made the following four experiments:—

4.38 grms. of bitartrate were mixed with .5 gm. of finely powdered pure Iceland spar and 50 c.c. of water. Carbonic acid was already evolved in the cold. The four mixtures were respectively boiled for 5, 10, 30, and 60 minutes; then at once neutralised.

After 5 minutes' boiling used		17.62 c.c. N S.
" 10	" "	17.12 "
" 30	" "	16.86 "
" 60	" "	16.00 "

If the calcic carbonate had fully acted, only 13.30 c.c. N S would have been required. In practice, the neutralising power of the calcic carbonate will chiefly depend on its state of division. The presence of calcic carbonate in tartars diminishes the yield of crystals, as insoluble calcic tartrate is formed, and the mother liquid contains uncrystallisable neutral potassic tartrate. This may be partially remedied by adding to the mother liquor a definite quantity of sulphuric acid, which will again form the bitartrate.

In the analysis of such a mixture it is the best thing to add, besides the potassic chloride, about 5 c.c. of acetic acid, which does not influence the accuracy of the process, as will be seen from Table VI.

2.5 grms. of bitartrate were dissolved in somewhat less than 55 c.c. of hot water; the liquid, after cooling, was diluted with glacial acetic acid exactly up to 55 c.c., and then mixed with 5 grms. of potassic chloride. After standing over night the crystals were washed 15 times with a 10 per cent. solution of potassic chloride (saturated with cream of tartar) and then titrated.

TABLE VI.

Temperature of fluid—		Acetic acid. c.c.	N Soda. c.c.	Bitartrate.	
Midnight.	At time of filtering.			Grms.	Per cent.
20°C.	23.5°C.	.50	13.06	2.4553	98.21
19	20	1.30	13.07	2.4572	98.29
..	21	4.75	13.05	2.4534	98.14
19.5	21	9.50	13.07	2.4572	98.29

If Verniere's proposals (treating the calcic tartrate with sulphuric acid, and making the tartaric acid thus liberated into cream of tartar by means of a potash salt) should find universal favour with the manufacturers of cream of tartar, the best plan will be to try and estimate as accurately as possible the total amount of real tartaric acid, and follow as closely as possible the actual process of manufacturing.

## THE CHEMISTRY OF TYROTOXICON, AND ITS ACTION UPON LOWER ANIMALS.\*

BY VICTOR C. VAUGHAN, M.D., PH.D.

SINCE making my last report on the investigations concerning the nature of tyrotoxin, I have continued my work, aided greatly by Messrs. F. G. Novy and E. V. Riker. We

\* From the *American Pharmacist*.



on ascertained that if some butyric acid ferment be prepared as is ordinarily done in the preparation of butyric acid, and some of this be added to normal milk, and the whole be kept in closely stoppered bottles for eight or ten days, the poison will be developed in the milk in considerable quantity. The milk should be filtered, the filtrate neutralised with sodium carbonate, and then extracted with ether.

Having a strong solution of the poison in absolute alcohol, which had been obtained from milk inoculated as stated above, we added to it some platinum chloride, and began to evaporate on the water-bath. As soon as the alcohol evaporated the residue exploded with great violence. The experiment was repeated a number of times with like results. From some of this alcoholic solution the platinum was removed with hydrogen sulphide gas; but the filtrate was then found to have lost its explosive property. This reminded us that diazobenzol compounds form with platinum chloride a highly explosive salt, and that diazobenzol is also decomposed by hydrogen sulphide.

Some diazobenzol was prepared according to the method of Griess (*Annalen der Chemie und der Pharmacie*, vol. 137), and comparisons made between this and tyrotoxinon. With equal parts of sulphuric acid and carbolic acid the prepared diazobenzol filtrate gave a green coloration, while with the same reagents tyrotoxinon gave a colour which varied from a yellow to an orange-red. But the diazobenzol nitrate dissolved in the whey of normal milk, and extracted with ether, or in the presence of other proteids, gave the same shades of colour as the tyrotoxinon did, and the potassium compound of the tyrotoxinon, prepared by the method to be given later, gives the green coloration. This colour test may be used as a preliminary test in examining milk for tyrotoxinon. It is best carried out as follows:—Place on a clean porcelain surface two or three drops each of pure sulphuric acid and pure carbolic acid. This mixture should remain colourless or nearly so. Then add a few drops of the aqueous solution of the residue left after the spontaneous evaporation of the ether. If tyrotoxinon be present an orange-red to a purple colour will be produced. This test is to be regarded as only a preliminary one, for it may be due to the presence of a nitrate or nitrite. The tyrotoxinon must be purified according to a method to be given farther on before the absence of a nitrate or nitrite can be positively demonstrated.

In the filtrate from milk which is rich in tyrotoxinon, after neutralization with sodium carbonate, filtration, and acidifying with hydrochloric acid, gold chloride produces a precipitate, which is insoluble in water, but soluble in hot alcohol, from which it separates on cooling in golden plates. Diazobenzol gives with gold chloride a precipitate having all these properties. In both cases the gold compound is decomposed by frequent treatment with hot alcohol, and this fact prevented any satisfactory ultimate analysis of the compound. It should be remarked here that from some samples of milk this gold salt obtained much more easily than from others, and the difference is dependent not so much upon the amount of tyrotoxinon present, as upon the condition of the other organic matter present. It is best obtained from samples which have stood in well stoppered bottles for a month or longer.

Thinking it not likely that the diazobenzol existed in the cheese and milk as a filtrate, we prepared some diazobenzol butyrate, and found the crystals of these to agree exactly with those of tyrotoxinon, and that they decomposed with like rapidity when exposed to moist air.

From tyrotoxinon obtained from milk, diazobenzol-potassium hydrate was obtained according to the method of Griess, and the per cent. of potassium in this compound was determined. The filtrate from the milk, which had been inoculated with the ferment, and kept in a stoppered bottle in a warm room for ten days, was neutralized with sodium carbonate, agitated with an equal volume of absolute ether, allowed to stand in a stoppered flask for twenty-four hours, the ether removed and allowed to evaporate from an open dish. The aqueous residue was acidified with nitric acid, then treated with an equal volume of a saturated solution of potassium hydrate, and the whole concentrated on the water-bath. On being heated the mixture became yellowish-brown, and emitted a peculiar aromatic odour. Both the colour and odour corresponded exactly with the colour and odour produced by carrying some of the artificial diazobenzol through a comparative test. On cooling the mass crystallised, the diazobenzol-potassium hydrate appearing in the test with the tyrotoxinon, and in the comparative test also, in beautiful six-sided plates, along with the prisms of potassium nitrate. The crystalline mass was treated with absolute alcohol, filtered, the filtrate evaporated on the water-bath, the residue dissolved in absolute alcohol, from which the diazobenzol-potassium hydrate was precipitated with ether. The precipitate was collected, washed with ether, dried, and the per cent. of potassium estimated as potassium sulphate. 0.2045 gram. of the diazobenzol-potassium hydrate yielded 0.109 gram. of potassium sulphate. Per cent. of potassium calculated, 24.42; found, 23.92.

Chemists will now appreciate the great difficulty that has been experienced in isolating the active agent of poisonous cheese. The readiness with which diazobenzol decomposes is well known. When warmed with water it breaks up into carbolic acid and nitrogen. Hydrogen sulphide decomposes it; therefore all attempts to obtain the poison by precipitating it with some base, such as mercury or lead, and then removing the base with hydrogen sulphide, have failed. Moreover, diazobenzol is only a transition product of putrefaction. I have frequently found that leaving some milk rich in the poison in an open beaker for twenty-four hours would be sufficient to destroy the whole of the poison.

The following experiments will show that the effects of tyrotoxinon and diazobenzol upon the lower animals are identical:—

*Experiment 1.*—From one half gallon of some milk which had stood in a tightly stoppered bottle for three months, there was obtained quite a concentrated aqueous solution of the poison, after the spontaneous evaporation of the ether. Ten drops of this placed in the mouth of a small dog three weeks old caused within a few minutes frothing at the mouth, retching, the vomiting of frothy fluid, rapid breathing, muscular spasm over the abdomen, and, after some time, watery stools. The next day the dog seemed to have partially recovered, but was unable to retain any food. This condition continuing for two days, the animal was killed with chloroform. No examination of the stomach was made.

*Experiment 2.*—Tyrotoxinon obtained from poisonous ice-cream was given to a cat. Within ten minutes the cat began to retch, and soon it vomited. The retching and vomiting continued for two hours, during which time the animal was under observation, and the next morning it was observed that the cat had passed several watery stools.



After this, although the cat could walk about the room, it was unable to retain any food. Several times it was seen to lap a little milk, but on doing so it would immediately begin to retch and vomit. This condition continuing, after three days the animal was placed under ether, and its abdominal organs examined. We certainly expected to find marked inflammation of the stomach; but we really did find the stomach and small intestines filled with a frothy serous fluid, such as had formed the vomited matter, and the mucous membrane very white and soft. There was not the slightest redness anywhere along the alimentary canal.

*Experiment 3.*—Some tyrotoxinon obtained from milk which had been inoculated with poisonous cream, and allowed to stand for forty-eight hours, was administered to a large old cat. It soon produced retching, but no vomiting or diarrhoea. The amount of the poison administered in this case was small.

*Experiment 4.*—Some tyrotoxinon from milk was given to a young, but full-grown cat. Within fifteen minutes there was marked and evidently painful retching, and within half an hour vomiting accompanied by rapid breathing. Later there were several stools, the first two of which contained fecal matter; but subsequent ones were rice-water like, and wholly free from fecal odour. After two days some more of the poison was given, and the vomiting and diarrhoea again induced. The animal was then anaesthetized, and examination of the stomach and intestine showed the mucous membrane blanched, as was found in experiment 2.

We have the records of a number of other experiments with tyrotoxinon on the lower animals; but as the symptoms induced in all were substantially the same, it is unnecessary to note them here. We will now give the effects observed in the lower animals after the use of the prepared diazobenzol.

*Experiment 5.*—Gave to a large old cat 100 milligrams. of diazobenzol butyrate. Immediately the animal began to purge. Then she lay upon the floor breathing rapidly and retching severely for two hours, when she died. The retching was most violent, but vomiting seemed impossible. *Post-mortem* examination showed the lungs greatly congested, but the mucous membrane of the stomach and intestine was not reddened. The stomach contained some food. I suppose that the congestion of the lungs was due to the violent retching.

*Experiment 6.*—To a young, but full-grown Maltese cat I gave 100 milligrams. of diazobenzol butyrate. With most violent retching, but without either vomiting or stool, the animal died within thirty minutes after the administration of the poison. The lungs were found acutely congested, and the stomach free from any redness. The circular fibres of the small intestine were tightly contracted.

*Experiment 7.*—Gave to a full-grown cat 25 milligrams. of diazobenzol butyrate. Within ten minutes vomiting and purging were induced. The first stools contained fecal matter; but the subsequent ones were like rice-water, and wholly free from fecal odour. After two days the cat was able to take food; then 10 milligrams. more of the poison were given, with the reproduction of the vomiting and purging. The animal then rapidly emaciated, and after a few days it was anaesthetized, and the mucous membrane of the stomach and intestine found blanched. The lungs were not congested.

*Experiment 8.*—10 milligrams. of the poison produced profuse diarrhoea, and continued vomiting in a cat.

*Experiment 9.*—75 milligrams. produced vomiting and diarrhoea with congestion of the lungs in a dog.

It seems unnecessary to detail any more of these experiments, as the identity of tyrotoxinon with diazobenzol is now established, not only by chemical analysis, but this proof is strengthened, if chemical analysis can be strengthened, by the action of the poison on the lower animals, and by the *post-mortem* appearance.

I think it highly probable that diazobenzol or some closely-allied substance will be found in all those foods which from putrefactive changes produce nausea, vomiting and diarrhoea. In some oysters which produced these symptoms I have recently found tyrotoxinon.

Milk or other fluid to be tested for this poison should be kept in well stoppered bottles; for if the fluid be exposed to the air the tyrotoxinon may decompose in a few hours. The filtrate from the milk, or the filtered aqueous extract of cheese should be neutralised with sodium carbonate, then shaken with half its volume of pure ether. Time should be given for the complete separation of the ether. Purified tyrotoxinon is insoluble in ether, and it probably owes its solubility in ether at this stage to the presence of impurities. After complete separation the ether should be removed by a pipette, and allowed to evaporate spontaneously from an open dish. The residue from the ether may be dissolved in distilled water, and again extracted with ether. To a drop of an aqueous solution of the ether residue apply the preliminary test with sulphuric and carbolic acid. To the remainder of the aqueous solution of the ether residue add an equal volume of a saturated solution of caustic potash, and evaporate the mixture on the water-bath. The double hydrate of potassium and diazobenzol will be formed if tyrotoxinon be present, and this may be recognised by its properties and reactions which have already been described.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

**ESTIMATION OF FAT IN MILK.**—MORSE, PIGGOT, AND BURTON.—This method consists in the dehydration of the milk by means of anhydrous sulphate of copper; the extraction of the fat by means of the low-boiling products of petroleum; the saponification of the butter by means of an excess of a standard solution of potassium hydrate in alcohol; and the determination of the excess of the alkali by means of a solution of hydrochloric acid. The following apparatus and reagents are required:—

- (1) A porcelain mortar and pestle.
- (2) An extraction tube, 14 or 15 mm. in diameter, 220 mm. in length, with funnel-shaped top. A straight chloride of calcium tube may be used for this.
- (3) A 200 c.c. Erlenmeyer flask, strong enough to be used with a filter pump.
- (4) A suitable stand for holding the flask and extraction tube.
- (5) Ten-cubic centimetre pipettes.
- (6) Weighing-glasses with ground-glass stoppers.
- (7) A low-boiling gasoline, distilling between 30° and 60°.
- (8) Dehydrated sulphate of copper.
- (9) Semi-normal solution of potash in 95 per cent. alcohol.
- (10) A semi-normal solution of hydrochloric acid.

*Manipulation.*—Place about 20 grams. of the anhydrous copper sulphate, roughly measured in a copper spoon of the size to hold about that amount, in a porcelain mortar;



a cavity in the centre of the mass with the pestle. Allow 10 c.c. of the milk to be added to the copper sulphate, being careful that none of it touches the sides of the flask. When the milk is nearly dry, grind the mass up with a little clean sand, transfer to the extraction tube, gently pressing it down in the tube by means of a glass rod. The lower portion of the extraction tube to be packed with clean cotton wool. The fat is then extracted in the following way: 15 c.c. of benzene is poured over the material in the extraction tube and drawn down with the aid of the filter pump, until the whole of the material to be extracted has become wet with the liquid, when the connection with the pump is closed; after about five minutes another portion of 15 c.c. of benzene is poured into the tube and the whole of the liquid slowly drawn through with aid of the pump into the flask. Usually one extraction of this kind is sufficient to withdraw the whole of the fat, but for the sake of greater accuracy the process may be repeated two or three times.

*Estimation.*—The benzene may be evaporated and the residual butter fat saponified with 25 c.c. of the approximately semi-normal potash. The residual alkali is determined by the addition of a few drops of the semi-normal hydrochloric acid, using phenol-phthalein as indicator. The difference between the amount required in this process and the amount necessary to saponify the quantity of alkali taken gives the amount of alkali required for the saponification. The number of milligrams of potash required for one gram. of the fat is about 230. The fat may also be accurately titrated without evaporating the benzene.

W. H. D.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

**ANALYSIS OF POTASSIUM-ANTIMONIC FLUORIDE.** By G. STEIN. *Chem. Zeit.*, 84. This double salt is now extensively used in dye-works, instead of tartar emetic, on account of its low price. To qualitatively test for its purity, the author adds potassium cyanide, which should give no blue colour, and baric chloride, with a little hydrochloric acid, should give no turbidity. It is, of course, better to make a quantitative analysis, which is easily performed, as follows:—5 gm. of the compound is dissolved in water, and after adding a few drops of phenol-phthalein, rendered just alkaline with ammonia. The precipitated antimonious oxide is collected on a weighed filter, washed, and dried at 110° C., and weighed. The yield should be 66 per cent. L. DE K.

**REACTIONS OF OAK-BARK TANNIN.** C. BÖTTINGER. *Liebigs Ann.*, 240.—The assay materials are still so unsatisfactory that everything which throws light on the subject must be considered welcome. The author has found the true oak-bark tannin to differ from bromine in a different way from other tannins, in so far as it yields a brownish deposit,  $C_{19}H_{14}Br_2O_{10}$ , which shows the following reactions: It is with difficulty soluble in water, but readily so in a mixture of alcohol and acetic ether. This solution with cupric sulphate, a precipitate; with ferro and ferricyanides, a green turbidity; with ferric chloride, the usual blue black. Alkalies soon dissolve it. It explodes when fused with solid potassic hydrate. When again heated with anhydrous bromine it reacts with effervescence into  $C_{19}H_{10}Br_4O_{10}$ , a reddish compound, which is easily soluble in alcohol, carbon disulphide, acetic acid, alkalies, and ammonia. L. DE K.

**AMMONIAC DITHIOCARBAMATE AS A REAGENT.** J. KLEIN. *Report Anal. Chemie*, 42, 1896. The action of carbon disulphide on an alcoholic solution of ammonia produces not only ammoniac dithiocarbamate, but also the sulpho-carbonate. To obtain the first in the state Mulder's process is the best. Ammonia gas is evolved from a mixture of

150 parts of sal ammoniac and 300 parts of quicklime, and passed into 600 parts of strong alcohol. Finally 95 parts of carbon disulphide are added. When the temperature is kept at 30° C only the dithiocarbamate will crystallise out, which must be washed with a little strong spirit, and dried between blotting paper. If after exposure some particles turn reddish, these must be rejected. To see if the salt is really free from sulphocarbonate add a little to an ammoniacal solution of nickel sulphate when no red colour should develop. The salt keeps far better as a 5 per cent. watery solution than in the solid state. As will be seen, it may be conveniently used instead of sulphuretted hydrogen, for the separation of many metals. Action on copper:—A solution of a copper salt, which must contain free HCl is completely precipitated on boiling, cupric dithiocarbamate being precipitated as a yellow powder which may be washed and collected without loss. The author prefers to ignite it in a current of hydrogen, and so form cuprous sulphide, which is then weighed. Iron, manganese, nickel, cobalt, alkaline earths and alkalis are not affected. Zinc comes down slightly with the copper, but may be removed by a second precipitation. This double precipitation is, in fact, always advisable when the copper is present in relative small quantities. In applying the process to brass, dissolve the alloy in nitric acid, evaporate with hydrochloric acid to dryness, dissolve residue in water containing about 5 per cent. HCl, boil and add the reagent. The precipitated copper is, after washing, redissolved in nitric acid and once more similarly treated. Action on zinc, etc.:—Zinc is completely precipitated on boiling if the liquid contains no free hydrochloric acid; free acetic acid, if not present in too large amount, does not prevent the precipitation. The author therefore recommends the addition of a little sodic acetate. The precipitate may be collected and washed without loss, and is, like the copper, best made into zinc sulphide before weighing. Ferric salts are reduced to ferrous salts, which are thrown down from a neutral solution. Nickel, cobalt, and manganese are but incompletely precipitated, even from an ammoniacal solution. Aluminium and chromium:—Aluminic sulphate is thrown down on boiling, probably as hydrate, with evolution of sulphuretted hydrogen. Chrome alum gives a precipitate, partly consisting of hydrate, partly of a blue body of a complex nature. The filtrate is also coloured blue. In presence of free acid chromates are reduced to chromic salts. Mercury, lead, silver, bismuth, cadmium, all yield characteristic precipitates. Cadmium may be separated from copper by first adding excess of ammonia and potassic cyanide, then an excess of the dithiocarbamate. On heating the cadmium is precipitated. Copper and mercury may also be similarly separated, using soda ley instead of ammonia. The copper may be recovered from the filtrate by adding hydrochloric acid. Tin and antimony also yield characteristic precipitates, but the author attaches great importance to the power of the reagent to precipitate arsenic, as there is no doubt it can be got absolutely free from arsenical impurities.

L. DE K.

## BOOKS, &amp;c., RECEIVED.

AMERICAN Analyst; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Druggist's Circular; Hospital Gazette; The Illustrated Sidney News; Independent Journal; Invention; Journal of the American Chemical Society; Journal of Microscopy and Natural Science; Justus Liebig's Annalen der Chemie; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; National Druggist; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Repertorium der Analytischen Chemie; San Francisco News Letter; Scientific American; Society of Arts Journal.

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# THE ANALYST.

FEBRUARY, 1888.

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(All Abstracts and Reviews deferred till next month owing to pressure of Society's matter.)

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Annual Meeting of the Society was held at Burlington House on Wednesday, the 11th January, the President, Mr. Allen, in the chair.

On the minutes of the previous meeting being read, Mr. Johnstone called attention to a statement made at the last meeting to the effect that he was unwell, which, he said, was made without authority, and was due to a misunderstanding, as he had not been unwell but only unable to attend the last meeting. The minutes were then confirmed.

Mr. JOHNSTONE then presented the accounts for the past year, audited by himself and Mr. Fox, showing a balance at the banker's of £151 9s. 1d.

Mr. ADAMS thereupon moved, and Mr. Dyer seconded, that £100 be invested in some Government funds or British Railway debentures; which was carried unanimously.

On Mr. ADAMS rising to propose the resolution of which he gave notice at the December meeting, as reported in THE ANALYST for January, respecting the appointment of Mr. F. Low, as Solicitor of the Society,

Mr. JOHNSTONE rose to a point of order, as the motion had been ordered to be on the agenda for that evening, and it was not there.

The PRESIDENT said it was certainly an omission, but, unless Mr. Johnstone could show a rule to the contrary, he should hold that Mr. Adams was entitled to proceed with his motion, of which due notice had been given and published in their journal.

Mr. JOHNSTONE said he merely wished to call attention to the irregularity.

Mr. ADAMS then moved, "That Mr. F. Low, of 17, King St., Cheapside, be appointed Solicitor to the Society, and that whenever legal business is likely to come before the Council he be requested to attend."

After considerable discussion Mr. ALLEN explained the reasons for appointing a solicitor; he said they were continually suffering in various parts of the country having cases defended by actual barristers, or by solicitors who had great practical experience in such matters, while the only person to represent the interest of the prosecutorial sanitary inspector, who had no weight with the magistrates, and, in cases where there was a dispute, was incapable of showing that previous cases had been decided in a particular way. It was thought to be desirable that they should have some man who would be a point of mastering the cases which had arisen under the Sale of Food Act, and who could, if necessary, be called on to undertake the prosecution of any cases which occurred, and most of them had known of cases where such assistance would have been desirable. It was proposed to give Mr. Low some sort of official position, and, as legal adviser of the Society, he would be invited to attend their Council meetings. There was no responsibility on the part of the Society involved in the matter. The Society would have nothing to do with any payment to him, as the local board might employ him would have to arrange that.

Mr. ADAMS said that Mr. Low was the son of a county magistrate, and the nephew of a barrister, and practised himself in the heart of the city of London. He considered him a very competent man, and one who was particularly eligible for the appointment. Turning from that, he would point out how essential such an appointment soon would be. The necessity for it had occurred to him in his own practice. There was no one they could employ, although the county authorities wished to do so, and gave him a *blank cheque*; but there was no one but a mere inspector. He (Mr. Adams) was a solicitor, who turned out to be a regular dummy, and those who had been placed in that position would know what a great advantage it would be to be able to put a finger at a moment's notice on a man who had made a study of the matter, and who had facilities afforded him for seeing the various processes carried out.

The question was still further discussed.

Mr. JOHNSTONE stating that no one outside the Council knew anything about the matter except those who were present at the last meeting.

Mr. DYER said he was not at all sure that the Council could not have decided the question themselves. It had been discussed at considerable length at one Council meeting and referred to a Sub-Committee; that Committee had reported to the Council meeting, and, more as a matter of form than anything else, it now came before the general body of the members.

Ultimately, at the suggestion of Mr. KINGZETT, Mr. Adams altered the terms of the motion, so that it should read, "That Mr. F. Low be appointed solicitor to the Society in an honorary capacity," and on this being put to the meeting it was carried unanimously.

The PRESIDENT then delivered his annual address as follows:—

#### PRESIDENT'S ADDRESS.

IN accordance with the custom of my predecessors in this chair, at the conclusion of each year of office I present to you a short *résumé* of the past and present position of the Society.



In the first place, I think we may congratulate ourselves on the position of the Society, both numerically and financially. During the year we have elected sixteen new members and eight new associates, while, as a set-off against this increase, there are ten members and five associates who have either resigned or been removed from the list of the Society for non-payment of their subscriptions. It is pleasant to have to record that during the past year no death has occurred among the subscribers to the Society. The numerical progress made by the Society is shown by the following figures:—

	1885.	1886.	1887.
Honorary Members	10	10	10
Ordinary Members	149	152	158
Associates ..	22	25	28

Financially, the Society also shows an improvement, the balance in hand being, I am informed, about £150, as against £100 on the occasion of the annual meeting last year.

Not only has the Society made satisfactory progress numerically and financially, but the number of interesting and important papers have been such as fully to maintain the standard of previous years. It is indeed encouraging, and a striking evidence of the vitality of the Society, that the number of papers contributed during the year has been as high as thirty, many of which, as will be seen by the following list, were of a useful and important character.

2.	"Arsenical Glauber's Salt" .. .. .	Charles Cameron
	"Estimation of Methyl-Alcohol" .. .. .	Otto Hehner
	"Poivrette" .. .. .	J. Campbell Brown
	"Composition of Milk and Milk Products" .. .. .	P. Vieth
	"Determination of Glycerin in Fats" .. .. .	Otto Hehner
	"Pavy Sugar Test" .. .. .	S. Stokes
	"Additional Note on Poivrette" .. .. .	J. Campbell Brown
9.	"Further Experiments on Volatility of Glycerin" .. .. .	Otto Hehner
	"Experiences of Curious Impurities, Admixtures, and Substitutions" .. .. .	A. H. Allen
13.	"Admixture of Starch with Yeast" .. .. .	W. F. H. Stock
	"Testing Skim Milk by the Lactometer" .. .. .	Harold Faber
	"Another New Pepper Adulterant" .. .. .	J. Campbell Brown
	"On Flitwick Water" .. .. .	W. Johnstone
1.	"An Improved Method of Detecting Quassia and Certain Other Hop-Substitutes in Beer" .. .. .	A. H. Allen
	"Note on the Composition of Some Preparations Sold as Hop-Substitutes" .. .. .	A. H. Allen and W. Chattaway
	"On the Beer and Butterine Bills" .. .. .	Otto Hehner
8.	"Notes on the Logwood Test for Alum in Bread" .. .. .	W. C. Young
	"Quantitative Determination of Pepper Mixtures" .. .. .	A. W. Stokes
	"Pepper" .. .. .	T. Stevenson
	"On the American Methods of Manufacturing Oleomargarine, and the Laws Recently Framed on the Subject in the United States" .. .. .	J. Muter
12.	"Examination of Wort" .. .. .	E. W. T. Jones
	"Estimation of Starch" .. .. .	

Nov. 9.	" Allowance for Decomposed Milk "	A. W. Stokes
"	" Preservation of Milk Samples "	Otto Hehner
"	" Substitution of Asbestos Cloth for Blotting-Paper "	W. Johnstone
"	" Does Neutral or Sub-Acetate of Lead Precipitate Hop Bitters? "	W. Johnstone
"	" Filtration "	Hehner & Richmond
"	" Alumina as a Natural Constituent of Wheat-Flour "	W. C. Young
Dec. 23.	" Estimation of Peroxide of Hydrogen "	C. Kingzett
"	" Relation Between Specific Gravity, Fat, and Solids not Fat, on the Basis of the Society's Process for Milk Analysis "	Hehner & Richmond

In addition to the formal papers in the foregoing list, highly interesting and important discussions have taken place in connection with the proposed legislation respecting Beer and Butter-Substitutes, and it is satisfactory to know that the Society was able to make its influence felt, and that its interests were directly represented before the Select Committee on Butter-Substitutes, which sat during last Parliamentary session. As a consequence, we may congratulate ourselves that the Margarine Act, which became law on the first of this month, is a far more satisfactory working measure than appeared probable from either of the Bills brought in. The clause introduced on the suggestion of Dr. James Bell, enabling an inspector to take any un-labelled sample of what is apparently butter without previously going through the form of purchase, appears likely to be of great value in practice, and it is to be hoped the same principle will be extended to certain other articles whenever a general amending Act is passed. That the passing of such an amending Act is highly desirable will be admitted on all sides, and, in my opinion, the time is ripe for us to take all the steps in our power to urge the Government to undertake the requisite legislation.

Circumstances have greatly changed since the Public Analysts were first appointed under the Adulteration Act of 1872; and even when the Parliamentary Committee sat in 1874 the Society of Public Analysts had no existence, and there were no means of taking combined action or making their wants felt. Furthermore, in 1872 there were no generally recognised methods of examining food, and many of the published processes were wholly untrustworthy. Even where properly qualified chemists were appointed as analysts, they were severely handicapped by the general want of a medium for exchanging ideas, and unfortunately in too many cases sanitary authorities insisted that the medical officers of health who were appointed contemporaneously, many of whom were perfectly innocent of a knowledge of chemical analysis, should also undertake the duties of food analysts. Now, however, by a process of natural selection and survival of the fittest, the Public Analysts of the country are a body of highly trained and eminently competent men. By individual industry and combined efforts exercised through the medium of this Society, unassisted by Government grants or patronage, and without either much aid or sympathy in quarters where they might fairly have expected it, they have mastered, one by one, some of the most difficult problems of food analysis, and it has now become an everyday practice to analyse, with sufficient practical accuracy, articles of food which seemed at first to defy all efforts. In 1872, and for some years afterwards, the mere detection, to say nothing of the determination, of foreign fatty matters in butter with sufficient certainty to justify proceedings against the vendor, appeared an almost hopeless problem; but, thanks to the ingenuity of Hehner and



gell, supplemented by the industry of Dupré, Muter, Jones, James Bell, and other chemists in England, of Koettstorfer and Reichert in Germany, and Caldwell, Waller, and other chemists in America, it is now possible to detect and estimate very minute proportions of adulterants in butter. Our methods of examining tea, coffee, starch, and other groceries have similarly improved, while the beneficial effect of the enactment of the Act will be seen in the fact that adulteration of these articles is far less common than was formerly the case. The adulteration of flour and bread by alum is another practice which may still linger in certain districts, but bids fair soon to become almost obsolete. Whatever may have been the practice formerly, the experience of public analysts of late years shows that the vile adulterations said to be practised in the case of spirits and other alcoholic liquors are now simply historical. In the case of beer, a Public Analyst can do little so long as there is no legal definition of its nature; but we have serious reason to complain that a clause should have been smuggled into the Customs and Inland Revenue Act of 1885, which was essentially a prohibition against watering of beer by brewers after fermentation, or by dealers and retailers of beer. This clause, which was practically an Amending Act, so far as related to the adulteration of beer, was passed without regard to the existence of Public Analysts, and provided for the detection of adulteration by the Inland Revenue Officers. It is only now that the public should know that public analysts were not concerned in and had no power to make such proceedings as those which have been common in London during the past year, according to which it is contended by the Inland Revenue that it is illegal and a fraud on the Revenue to mix together two different kinds of beer, although each of them has already paid duty according to its character.

But the gravest disadvantage under which public analysts linger, and the direction in which an alteration of their present position is most urgent, consists in the present regulations respecting the reference of milk and other perishable articles to the chemists at Somerset House. This matter has been so recently and fully discussed that it is unnecessary for me to refer to it at length, but I cordially endorse the opinion expressed at the recent meeting of the Society, that the time for mere talk is at an end, and that the analysts should take formal action by a memorial addressed to, or deputation laying upon, the proper authorities. So long as we content ourselves with simply complaining, so long the present condition of affairs is likely to continue; but I believe there is now such an overwhelming mass of evidence of the failure of justice under the present system that the authorities would feel bound to take action of some sort if the matter were brought formally before them.

Dr. VIETH proposed a vote of thanks to the President for his services during the year. He had made a great sacrifice of time and had taken a great deal of trouble in their affairs, having attended every single general and council meeting during the year. The vote having been carried unanimously, the PRESIDENT returned thanks and received a vote of thanks to the officers of the Society for their services during the year. The President also proposed a vote of thanks to the Council of the Chemical Society for the use of their rooms.

On the ballot papers being opened it was announced that the following were elected Officers and Council of the Society for 1888:—

*President*.—A. H. Allen, F.C.S., F.I.C.

*Vice-Presidents* (who have filled the office of President).—A. Dupré, Ph.D., F.C.S., F.I.C.; C. Heisch, F.C.S., F.I.C.; Alfred Hill, M.D., F.C.S., F.I.C.; Ph.D., M.A., F.R.S.E., F.C.S., F.I.C. (Who have not filled the office of President).—S. Harvey, F.C.S.; P. Vieth, Ph.D., F.C.S.; C. R. Alder Wright, D.Sc., F.R.S., F.I.C.

*Treasurer*.—C. W. Heaton, F.C.S., F.I.C.

*Hon. Secretaries*.—Bernard Dyer, B.Sc., F.C.S., F.I.C.; Otto Hehner, F.I.C.

*Other Members of Council*.—J. Baynes, F.C.S., F.I.C.; J. A. R. Newland, F.I.C.; E. Seaton, M.D., F.R.C.P.; W. J. Sykes, M.D.; J. A. Voelcker, Ph.D., F.C.S.

The names of those Members of Council whose term of office has not yet expired and who consequently do not retire this year, are M. A. Adams, F.R.C.S., F.I.C.; Fox, F.C.S.; C. N. Hake, F.I.C.; C. T. Kingzett, F.C.S., F.I.C.; W. Boverton, F.C.S., F.I.C.; W. F. K. Stock, F.C.S., F.I.C.; R. R. Tatlock, F.R.S.E., F.I.C.

The following gentlemen were also elected:—

As Members: Laurence Briant, analyst; John Heron, F.C.S., F.I.C.; W. E. Matthews, Melbourne, public analyst.

As Associate: E. Michael, assistant to Mr. Stokes.

The President read a paper on "Laboratory Notes."

The Annual Dinner was afterwards held at the Criterion Restaurant, when a very pleasant and convivial evening was spent, notwithstanding the cold which prevailed outside.

The next meeting of the Society will be held at Burlington House on the 8th inst.

## ON THE RELATION OF SPECIFIC GRAVITY, FAT AND SOLIDS IN MILK, UPON THE BASIS OF THE SOCIETY OF PUBLIC ANALYSTS METHOD.

BY OTTO HEHNER AND H. DROOP RICHMOND.

(Read before the Society on December 14th, 1887.)

It will be within the memory of some members of the Society that a few years ago we worked out a formula which expressed the relation between specific gravity and solids-not-fat in milk (ANALYST, vol. vii., p. 129), upon the basis of the process, which was then still in general use among analysts. The formula did not claim to originality, for both Behrend and Morgen, and Clausnitzer and previously given formulae intended to express that relation. It, however, attracted the attention of public analysts to the fact that there was such a relation, which was by no means generally recognised.

The Wanklyn process of direct extraction of fat from the milk solid



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apply, a matter of the past, and the Adams process of fat extraction from the paper oil being generally recognised, and adopted by the Society's Milk Committee, and analysts in foreign countries, it appeared very desirable to work out a formula for the relation of specific gravity, fat, and solids-not-fat upon the bases of Mr. Adams' method of extraction. To every particular process of milk analysis appertains a particular relation or formula. Morgen's plaster extraction formula is not applicable to the Wanklyn method, nor our Wanklyn formula to the coil process. This fact cannot be too strongly impressed, and has not unfrequently been overlooked by critics of the different formulae.

We have, therefore, taken upon ourselves to establish a mathematical expression of this relation for the coil process.

The method pursued was not precisely that recommended by the Society of Public Analysts, but a slight modification, which simplifies the process. 5 c.c. of milk were weighed into a weighed dish, and the weight accurately ascertained. From these the total solids were estimated by evaporation and drying to practical constancy. 5 c.c. of the same milk were then dropped as equally as possible upon a strip of demy blotting paper of regulation size, the paper being pinned at one of the small edges against a frame on a bench, and the lower end folded up for about half an inch, to catch any drops which might otherwise run down and be lost. The weight of the 5 c.c. was taken to be the same as that previously ascertained. Any error which could thus occur could amount to only a milligramme or two, and would be without appreciable influence upon the result. The strips of paper were allowed to hang in the air until they were practically dry, which took generally about one hour. They were then coiled up and extracted with ether in a Soxhlet tube for at least an hour and a half. We carefully avoided drying the paper at elevated temperatures, in order to reduce any possible oxidation of the oil to a minimum. The extraction should last an hour and a half, as we have convinced ourselves that a less time is frequently insufficient. The paper coils were not previously extracted with ether, an allowance (which amounted to '0120 grm. for each) being made for soluble matter contained in each coil, based upon careful blank experiments.

The process thus simplified (after the example of Mr. Allen and Mr. W. Thomson) shows a saving of a number of weighings over the method of the Milk Committee, and further raises than diminishes the accuracy, all chance of evaporation during the absorption by the coil, and subsequent influence upon the result, being thus avoided.

The specific gravities were taken by a Spengel tube with very narrow capillaries, or with an improved Westphal balance of great delicacy, procured from Messrs. Sartorius, in Göttingen.

The following series of milk analyses embraces milks of all descriptions. There are normal, skimmed, skimmed and watered milk, cream, milk enriched with cream, and the same watered. We hope that no reproach can be made that the series is not a thoroughly representative one. Many of the results are the averages of two self-agreing duplicate determinations.

In column 1 of the table are the specific gravities, marked "G," being the figures beyond 1,000; in column 2 the total solids per cent; in column 3 the fat per cent.

TABLE OF ANALYSES.

	1	2	3	4	5	6	7	8
	G	T	F (found)	F (calc. from formula)	Diff.	F (calc. with correction)	Diff.	F & M
1	26.1	10.23	3.07	3.08	+ .01	3.08	+ .01	2.88
2	33.1	14.94	5.64	5.62	- .02	5.62	- .02	5.34
3	28.7	11.69	3.39	3.78	- .11	3.78	- .11	8.52
4	24.2	10.62	3.91	3.84	- .07	3.84	- .07	3.61
5	23.8	11.08	4.24	4.32	+ .08	4.32	+ .08	4.08
6	31.3	12.40	3.77	3.82	+ .05	3.82	+ .05	3.50
7	31.0	12.22	3.65	3.74	+ .09	3.74	+ .09	3.36
8	23.1	8.53	2.27	2.28	+ .01	2.27	—	2.09
9	32.0	13.13	4.39	4.30	- .09	4.30	- .09	4.05
10	31.0	13.40	4.78	4.75	- .03	4.75	- .03	4.50
11	33.4	11.75	2.79	2.81	+ .02	2.79	—	2.62
12	29.7	11.20	3.28	3.14	- .14	3.13	- .15	2.89
13	30.4	11.69	3.53	3.41	- .12	3.41	- .12	3.19
14	32.9	14.13	5.05	4.95	- .10	4.95	- .10	4.71
15	30.6	14.07	5.43	5.41	- .02	5.41	- .02	5.13
16	32.3	13.86	5.01	4.86	- .15	4.86	- .15	4.60
17	28.4	11.90	3.86	4.03	+ .17	4.03	+ .17	3.79
18	30.6	14.03	5.47	5.38	- .09	5.38	- .09	5.14
19	26.8	11.00	3.49	3.60	+ .11	3.60	+ .11	3.37
20	32.9	13.05	3.89	4.03	+ .14	4.03	+ .14	3.81
21	32.4	12.71	3.75	3.81	+ .06	3.81	+ .06	3.59
22	31.2	15.23	6.20	6.28	+ .08	6.28	+ .08	5.96



TABLE OF ANALYSES—*continued*.

1		2	3	4	5	6	7	8	Diff.
G		T	F (found)	F (calc. from formula)	Diff.	F (calc. with correction)	Diff.	F & M	
23	32.4	12.41	3.51	3.59	+ .08	3.58	+ .07	3.37	— .14
24	31.3	11.10	2.71	2.71	—	2.69	— .02	2.52	— .19
25	32.8	13.03	4.10	4.04	— .06	4.04	— .06	3.80	— .30
26	32.2	12.36	3.62	3.59	— .05	3.58	— .04	3.37	— .25
27	32.7	12.91	4.05	3.95	— .10	3.95	— .10	3.72	— .33
28	32.6	13.05	4.16	4.10	— .06	4.10	— .06	3.86	— .30
29	32.5	13.07	4.22	4.14	— .08	4.14	— .08	3.89	— .33
30	31.4	13.00	4.32	4.31	— .01	4.31	— .01	4.08	— .24
31	30.0	14.35	5.82	5.78	— .04	5.78	— .04	5.49	— .33
32	10.2	30.79	24.06	24.23	+ .17	24.23	+ .17	23.41	— .65
33	35.6	9.52	.39	.41	+ .02	.35	— .04	.30	— .09
34	35.6	9.69	.43	.56	+ .13	.50	+ .07	.43	—
35	33.3	9.21	.57	.64	+ .07	.58	+ .01	.52	— .05
36	26.4	7.21	.23	.43	+ .20	.36	+ .13	.34	+ .11
37	23.8	19.65	11.63	11.68	+ .05	11.68	+ .05	11.22	— .41
38	12.0	10.17	5.98	6.12	+ .14	6.12	+ .14	5.85	— .13
39	35.4	9.49	.35	.43	+ .08	.37	+ .02	.29	— .06
40	32.6	12.80	4.03	3.88	— .15	3.88	— .15	3.65	— .38
41	28.2	11.36	3.65	3.61	— .04	3.61	— .04	3.38	— .27
42	31.6	12.88	4.26	4.17	— .09	4.17	— .09	3.94	— .32

Aver. 29.75

12.64

4.37

Probable error

± .080

± .075

— .26

Maximum error

.20

.17

.65

Hitherto all milk formulæ which have been constructed have been based upon the assumption that the specific gravity of the fat contained in milk was a known quantity. Thus Behrend and Morgen assumed that each per cent. of fat depressed the gravity by 1, whilst Fleischmann, in his new formula  $F = .833 T - 2.22 \frac{100S - 100}{S}$  takes the gravity of fat to be  $-.7$ . One of us, in his formula applying to the Wanklyn process, took the gravity of fat to be  $-.725$ , based upon results of Wigner and Blyth. But it is by no means certain that the gravity of the fat as contained in milk is equal to the gravity of the solid extracted milk-fat. The fat in milk is mostly in the fluid state, whilst separated butter-fat at  $60^{\circ} F$  is solid.

By the employment of the reasoning used by one of us in a former paper on this subject, *i.e.*, that each per cent. of fat would lower the gravity by a certain amount ( $f$ ), and each per cent. of solids not fat would raise the gravity by a certain amount ( $s$ ) we get the equation—

$$\begin{aligned} G &= Ss - Ff, \text{ or as total solids } (T) - F = S \\ G &= (T - F)s - Ff \\ \text{or } Ts - G &= F(s + f) \\ \text{or } \frac{F}{T} &= \frac{s}{s + f} - \frac{G}{T} \cdot \frac{1}{s + f} \text{ which is the formula to the straight line} \end{aligned}$$

on which all our results should lie if plotted with  $\frac{F}{T} + \frac{G}{T}$  as abscissae and coordinates respectively.

But every straight line may be expressed by the formula  $x = \frac{dy}{dx} \cdot y + \text{const.}$

$$\therefore \frac{\frac{F}{T}}{\frac{G}{T}} = - \frac{1}{s + f} \text{ which gives the value of } s + f, \text{ and substitution in equation } Ts - G = F(s + f)$$

gives us  $s$ , and consequently  $f$ ; by differentiation of our results the values—

$$\left. \begin{aligned} f &= .646 \\ s &= 3.938 \end{aligned} \right\} \text{ are obtained which give the equation } F = .859 T - .2186 G, \\ \text{or } T - .254 G = 1.164 F.$$

On plotting the results with  $\frac{F}{T}$  as abscissae, and  $\frac{G}{T}$  as coordinates, and drawing the line  $F = .859 T - .2186 G$ , it is seen, that whilst this line well expresses the relations of gravity and fat in average milks, it does not accurately apply to extreme cases, plainly indicating that a straight line cannot express the results, and that our initial assumption must be incorrect. This fact might have been foreseen had we taken into account the effect of dilution on (say) a solution of sugar, where it is well known that the law is not of a simple order, but is expressed by a parabolic curve; as our results were few at the ends of curve where the divergence from a straight line was most perceptible,



and the errors of experiment were large, we have not attempted to work out the actual curve, but have contented ourselves with making an approximation, which should be sufficiently near for all practical purposes ; this is—

$$F = .859 T - .2186 G - .05 \left( \frac{G}{T} - 2.5 \right) \text{ when } \frac{G}{T} \text{ exceeds } 2.5.$$

This, as will be seen by a glance at the diagram, and at the table of analyses, (columns 6 and 7,) very fairly expresses the results on the lower part of the curve, but we have decided to apply no correction to upper part, first, because nobody would think of analysing a cream in this way, and, second, because the errors of experiment are too large, and our results too few to enable us to correct with any degree of accuracy.

For average milks the formula  $F = \frac{5}{6} \left( T - \frac{G}{4} \right)$  gives results within about .02 of the other and is much easier to remember.

In the annexed diagram A is the straight line  $F = .859 T - .2186 G$

B expresses the correction  $- .05 \left( \frac{G}{T} - 2.5 \right)$

C represents Fleischmann's new formula

D is Hehner's old Wanklyn formula.

The difference between the three methods of milk analyses is thus well seen.

In column 8 of our table of analyses are given the results calculated from Fleischmann's new formula. On the average these are .26 per cent. lower than the results by the coil process. On applying the Wanklyn formula previously referred to, we find that on the average, the direct fat extraction gives results which are .45 per cent. lower than those by the coil. But in the case of very poor skim milk the Wanklyn formula actually gives negative results, showing that that method is quite inapplicable.

These calculated averages confirm in a remarkable degree the conclusions of the Milk Committee as to the relations of the results obtained by the various methods, namely, that the coil figures were .5 per cent., the plaster results from .2 to .3 per cent. lower than those by direct extraction.

From our formula the following tables have been calculated, giving at a glance the fat from total solids and specific gravity for milk of widely varying composition.

We would, in conclusion, strongly urge, that no milk analyses be accepted as correct which do not correspond closely with the calculated results. Reference to previous pages of the ANALYST, and to well known works on adulteration will prove how much has been sinned in this respect.

SOCIETY OF PUBLIC ANALYSTS' METHOD OF MILK ANALYSIS.  
FAT CALCULATED FROM SPECIFIC GRAVITY AND TOTAL SOLIDS—

$$F = .859 T - .2182 G - .05 \left( \frac{G}{T} - 2.5 \right)$$

if positive.

By OTTO HEHNER AND H. DROOP RICHMOND.

SPECIFIC GRAVITY.	TOTAL SOLIDS.									
	10.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
1024.0	3.35	3.43	3.52	3.61	3.70	3.78	3.87	3.95	4.04	4.13
1024.5	3.24	3.33	3.42	3.50	3.59	3.67	3.76	3.85	3.93	4.02
1025.0	3.13	3.22	3.31	3.39	3.48	3.56	3.65	3.74	3.82	3.91
1025.5	3.03	3.11	3.20	3.28	3.37	3.46	3.54	3.63	3.71	3.80
1026.0	2.92	3.01	3.09	3.17	3.26	3.35	3.43	3.52	3.60	3.69
1026.5	2.81	2.90	2.98	3.06	3.15	3.24	3.32	3.41	3.50	3.58
1027.0	2.69	2.78	2.87	2.95	3.04	3.12	3.21	3.30	3.39	3.47
1027.5	2.58	2.67	2.76	2.84	2.93	3.01	3.10	3.19	3.27	3.36
1028.0	2.47	2.55	2.64	2.73	2.81	2.90	2.99	3.07	3.16	3.25
1028.5	2.35	2.44	2.52	2.62	2.70	2.79	2.88	2.96	3.05	3.13
1029.0	2.24	2.33	2.41	2.51	2.59	2.68	2.77	2.85	2.94	3.02
1029.5	2.13	2.22	2.30	2.39	2.48	2.57	2.65	2.74	2.83	2.91
1030.0	2.02	2.11	2.20	2.28	2.37	2.46	2.54	2.63	2.72	2.81
1030.5	1.91	2.00	2.09	2.17	2.26	2.35	2.44	2.52	2.61	2.69
1031.0	1.80	1.89	1.97	2.06	2.15	2.23	2.33	2.41	2.49	2.58
1031.5	1.69	1.77	1.86	1.95	2.04	2.12	2.21	2.30	2.38	2.47
1032.0	1.58	1.67	1.75	1.84	1.93	2.01	2.10	2.19	2.27	2.36
1032.5	1.47	1.56	1.64	1.73	1.82	1.90	1.99	2.08	2.16	2.25
1033.0	1.36	1.45	1.53	1.61	1.70	1.79	1.88	1.97	2.06	2.14
1033.5	1.25	1.33	1.42	1.50	1.59	1.67	1.76	1.85	1.95	2.03
1034.0	1.14	1.22	1.30	1.39	1.47	1.56	1.65	1.74	1.83	1.91
1034.5	1.02	1.11	1.19	1.28	1.36	1.45	1.54	1.62	1.71	1.80
1035.0	.91	1.00	1.08	1.17	1.25	1.34	1.43	1.51	1.60	1.69



FAT CALCULATED—*continued.*

SPECIFIC GRAVITY.	TOTAL SOLIDS.									
	11·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
1024·0	4·21	4·30	4·38	4·47	4·56	4·64	4·73	4·81	4·90	4·97
1024·5	4·10	4·19	4·27	4·36	4·45	4·53	4·62	4·70	4·79	4·87
1025·0	3·99	4·08	4·17	4·25	4·34	4·42	4·51	4·59	4·68	4·78
1025·5	3·89	3·97	4·06	4·14	4·23	4·32	4·40	4·49	4·57	4·66
1026·0	3·78	3·86	3·95	4·03	4·12	4·21	4·29	4·38	4·46	4·54
1026·5	3·67	3·75	3·84	3·93	4·01	4·10	4·18	4·27	4·35	4·43
1027·0	3·56	3·64	3·73	3·82	3·90	3·99	4·07	4·16	4·24	4·32
1027·5	3·45	3·54	3·62	3·71	3·79	3·88	3·96	4·05	4·14	4·21
1028·0	3·34	3·43	3·51	3·60	3·68	3·77	3·85	3·94	4·03	4·10
1028·5	3·23	3·32	3·40	3·49	3·58	3·66	3·75	3·83	3·92	4·00
1029·0	3·12	3·21	3·29	3·38	3·47	3·55	3·64	3·72	3·81	3·89
1029·5	3·00	3·10	3·18	3·27	3·36	3·44	3·53	3·61	3·70	3·78
1030·0	2·89	2·98	3·07	3·16	3·25	3·33	3·42	3·50	3·59	3·67
1030·5	2·78	2·87	2·96	3·04	3·14	3·23	3·31	3·40	3·48	3·56
1031·0	2·66	2·76	2·85	2·93	3·03	3·12	3·20	3·29	3·37	3·45
1031·5	2·55	2·64	2·73	2·82	2·91	3·00	3·09	3·18	3·26	3·34
1032·0	2·44	2·53	2·62	2·71	2·80	2·88	2·97	3·06	3·14	3·22
1032·5	2·33	2·42	2·51	2·60	2·68	2·77	2·86	2·95	3·03	3·11
1033·0	2·22	2·31	2·40	2·49	2·57	2·66	2·74	2·83	2·92	3·00
1033·5	2·11	2·20	2·29	2·37	2·46	2·55	2·63	2·72	2·81	2·89
1034·0	2·00	2·09	2·18	2·26	2·35	2·44	2·52	2·61	2·70	2·78
1034·5	1·89	1·98	2·07	2·15	2·24	2·33	2·41	2·50	2·58	2·67
1035·0	1·78	1·87	1·95	2·04	2·13	2·22	2·30	2·39	2·47	2·56

FAT CALCULATED—*continued.*

SPECIFIC GRAVITY.	TOTAL SOLIDS.									
	12·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
1024·0	5·07	5·16	5·24	5·33	5·41	5·50	5·59	5·67	5·76	5·85
1024·5	4·96	5·05	5·13	5·22	5·31	5·39	5·48	5·56	5·65	5·74
1025·0	4·85	4·94	5·02	5·11	5·20	5·28	5·37	5·45	5·54	5·63
1025·5	4·74	4·83	4·91	5·00	5·09	5·17	5·26	5·34	5·43	5·52
1026·0	4·63	4·72	4·81	4·89	4·98	5·06	5·15	5·24	5·32	5·41
1026·5	4·52	4·61	4·70	4·78	4·87	4·96	5·04	5·13	5·21	5·30
1027·0	4·42	4·50	4·59	4·67	4·76	4·85	4·93	5·02	5·10	5·19
1027·5	4·30	4·39	4·48	4·57	4·65	4·74	4·82	4·91	4·99	5·08
1028·0	4·20	4·28	4·37	4·46	4·54	4·63	4·71	4·80	4·88	4·97
1028·5	4·09	4·18	4·26	4·35	4·43	4·52	4·60	4·69	4·78	4·86
1029·0	3·98	4·07	4·15	4·24	4·32	4·40	4·49	4·58	4·67	4·75
1029·5	3·87	3·96	4·04	4·13	4·21	4·30	4·39	4·47	4·56	4·62
1030·0	3·76	3·85	3·93	4·02	4·11	4·19	4·28	4·36	4·45	4·53
1030·5	3·65	3·74	3·83	3·91	4·09	4·08	4·17	4·25	4·34	4·42
1031·0	3·54	3·63	3·72	3·80	3·89	3·97	4·06	4·14	4·23	4·32
1031·5	3·43	3·52	3·61	3·69	3·78	3·86	3·95	4·04	4·11	4·21
1032·0	3·32	3·41	3·49	3·58	3·66	3·75	3·84	3·93	4·01	4·10
1032·5	3·20	3·30	3·38	3·47	3·55	3·64	3·73	3·82	3·90	3·99
1033·0	3·10	3·19	3·27	3·35	3·44	3·54	3·61	3·70	3·79	3·88
1033·5	2·98	3·07	3·16	3·24	3·33	3·42	3·50	3·59	3·68	3·76
1034·0	2·87	2·95	3·05	3·13	3·21	3·30	3·39	3·48	3·57	3·65
1034·5	2·76	2·85	2·93	3·02	3·10	3·19	3·28	3·37	3·46	3·54
1035·0	2·65	2·74	2·82	2·91	2·99	3·08	3·16	3·25	3·35	3·43



FAT CALCULATED—*continued.*

SPECIFIC GRAVITY.	TOTAL SOLIDS.									
	13·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
1024·0	5·93	6·02	6·10	6·19	6·27	6·36	6·44	6·53	6·62	6·70
1024·5	5·82	5·91	5·99	6·08	6·16	6·25	6·33	6·42	6·51	6·59
1025·0	5·71	5·80	5·88	5·97	6·06	6·14	6·23	6·31	6·40	6·48
1025·5	5·60	5·69	5·78	5·86	5·95	6·03	6·12	6·20	6·29	6·37
1026·0	5·49	5·58	5·67	5·75	5·84	5·92	6·01	6·09	6·18	6·27
1026·5	5·38	5·47	5·56	5·64	5·73	5·82	5·90	5·99	6·07	6·16
1027·0	5·28	5·36	5·45	5·53	5·62	5·71	5·79	5·88	5·96	6·05
1027·5	5·17	5·25	5·34	5·42	5·51	5·60	5·68	5·77	5·85	5·94
1028·0	5·06	5·14	5·23	5·31	5·40	5·49	5·57	5·66	5·74	5·83
1028·5	4·95	5·03	5·12	5·20	5·29	5·38	5·46	5·55	5·63	5·72
1029·0	4·84	4·92	5·01	5·09	5·18	5·27	5·35	5·44	5·52	5·61
1029·5	4·73	4·81	4·90	4·99	5·07	5·16	5·24	5·33	5·41	5·50
1030·0	4·62	4·71	4·79	4·88	4·96	5·05	5·14	5·22	5·31	5·39
1030·5	4·51	4·60	4·68	4·77	4·85	4·94	5·03	5·11	5·20	5·28
1031·0	4·40	4·49	4·57	4·66	4·75	4·83	4·92	5·00	5·09	5·18
1031·5	4·29	4·38	4·46	4·55	4·64	4·72	4·81	4·89	4·98	5·07
1032·0	4·19	4·27	4·36	4·44	4·53	4·61	4·70	4·79	4·87	4·96
1032·5	4·08	4·16	4·25	4·33	4·42	4·50	4·59	4·68	4·76	4·85
1033·0	3·97	4·05	4·14	4·22	4·31	4·40	4·48	4·57	4·65	4·74
1033·5	3·86	3·94	4·03	4·11	4·20	4·29	4·37	4·46	4·54	4·63
1034·0	3·74	3·83	3·92	4·00	4·09	4·18	4·26	4·35	4·44	4·52
1034·5	3·63	3·72	3·80	3·89	3·98	4·07	4·15	4·24	4·33	4·41
1035·0	3·52	3·61	3·69	3·78	3·87	3·96	4·04	4·13	4·22	4·30

## FAT CALCULATED—continued.

SPECIFIC GRAVITY.	TOTAL SOLIDS.											DIFFERENCE TABLE.
	14.0	.1	.2	.3	.4	.5	.6	.7	.8	.9	15.0	
1024.0	6.79	6.88	6.96	7.05	7.13	7.22	7.30	7.39	7.48	7.56	7.65	TOTAL SOLIDS.  FAT.
1024.5	6.68	6.77	6.85	6.94	7.02	7.11	7.19	7.28	7.37	7.45	7.54	
1025.0	6.57	6.66	6.74	6.83	6.91	7.00	7.08	7.17	7.26	7.34	7.43	
1025.5	6.46	6.55	6.63	6.72	6.80	6.89	6.98	7.06	7.15	7.23	7.32	
1026.0	6.35	6.44	6.52	6.61	6.70	6.78	6.87	6.95	7.04	7.13	7.21	
1026.5	6.24	6.33	6.41	6.50	6.59	6.67	6.76	6.84	6.93	7.02	7.10	
1027.0	6.13	6.22	6.31	6.39	6.48	6.56	6.65	6.74	6.82	6.91	6.99	
1027.5	6.02	6.11	6.20	6.28	6.37	6.46	6.54	6.63	6.71	6.80	6.88	
1028.0	5.92	6.00	6.09	6.17	6.26	6.35	6.43	6.52	6.60	6.69	6.77	
1028.5	5.81	5.89	5.98	6.06	6.15	6.24	6.32	6.41	6.50	6.58	6.66	GRAVITY.  FAT.
1029.0	5.70	5.78	5.87	5.95	6.04	6.13	6.21	6.30	6.39	6.47	6.56	
1029.5	5.59	5.67	5.76	5.84	5.93	6.02	6.10	6.19	6.28	6.36	6.45	
1030.0	5.48	5.57	5.65	5.74	5.82	5.91	5.99	6.08	6.17	6.25	6.34	
1030.5	5.37	5.46	5.54	5.63	5.71	5.80	5.89	5.97	6.06	6.14	6.23	
1031.0	5.26	5.35	5.43	5.52	5.61	5.69	5.78	5.86	5.95	6.04	6.12	
1031.5	5.15	5.24	5.32	5.41	5.50	5.58	5.67	5.75	5.84	5.93	6.01	
1032.0	5.04	5.13	5.22	5.30	5.39	5.47	5.56	5.64	5.73	5.82	5.90	
1032.5	4.93	5.02	5.11	5.19	5.28	5.36	5.45	5.53	5.62	5.71	5.79	
1033.0	4.83	4.91	5.00	5.08	5.17	5.26	5.34	5.42	5.51	5.60	5.68	+1 = -.02 +2 = -.04 +3 = -.07 +4 = -.09
1033.5	4.72	4.80	4.89	4.97	5.06	5.15	5.23	5.32	5.40	5.49	5.57	
1034.0	4.61	4.69	4.78	4.86	4.95	5.04	5.12	5.21	5.29	5.38	5.47	
1034.5	4.50	4.58	4.67	4.75	4.84	4.93	5.01	5.10	5.18	5.27	5.36	
1035.0	4.39	4.47	4.56	4.64	4.73	4.82	4.90	4.99	5.08	5.16	5.25	

(Conclusion of the Society's Proceedings.)



## MARGARINE ACT, 1887.

Act for the better prevention of the fraudulent sale of margarine.

Whereas it is expedient that further provision should be made for protecting the public against the sale as butter of substances made in imitation of butter, as well as of butter mixed with any such substances:—

Be it therefore enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:—

[*Short Title.*] 1. This Act may be cited as the Margarine Act, 1887.

[*Commencement of Act.*] 2. This Act shall come into operation on the first day of January one thousand eight hundred and eighty-eight.

[*Definition.*] 3. The word "butter" shall mean the substance usually known as butter, made exclusively from milk or cream, or both, with or without salt or other preservative, and with or without the addition of colouring matter.

The word "margarine" shall mean all substances, whether compounds or otherwise, prepared in imitation of butter, and whether mixed with butter or not, and no such substance shall be lawfully sold, except under the name of margarine, and under the conditions set forth in this Act.

[*Penalty.*] 4. Every person dealing in margarine, whether wholesale or retail, whether a manufacturer, importer, or as consignor or consignee, or as commission agent or otherwise, who is found guilty of an offence under this Act, shall be liable on summary conviction for the first offence to a fine not exceeding twenty pounds, and for the second offence to a fine not exceeding fifty pounds, and for the third or any subsequent offence to a fine not exceeding one hundred pounds.

[*Exemption from penalty.*] 5. Where an employer is charged with an offence against this Act he shall be entitled, upon information duly laid by him, to have any other person whom he charges as the actual offender brought before the Court at the time appointed for hearing the charge, and if, after the commission of the offence has been proved, the employer proves to the satisfaction of the Court that he had used due diligence to enforce the execution of this Act, and that the said other person had committed the offence in question without his knowledge, consent, or connivance, the said other person shall be summarily convicted of such offence, and the employer shall be exempt from any penalty.

[*Marking of cases.*] 6. Every person dealing in margarine in the manner described in the preceding section shall conform to the following regulations:—

Every package, whether open or closed, and containing margarine, shall be branded or durably marked "Margarine" on the top, bottom, and sides, in printed capital letters, not less than three quarters of an inch square; and if such margarine be exposed for sale, by retail, there shall be attached to each parcel thereof so exposed, and in such manner as to be clearly visible to the purchaser, a label marked in printed capital letters not less than one and a half inches square, "Margarine"; and every person selling margarine by retail, save in a package duly branded or durably marked as aforesaid, shall in every case deliver the same to the purchaser in or with a paper wrapper, on which shall be printed in capital letters, not less than a quarter of an inch square, "Margarine."

[*Presumption against vendor.*] 7. Every person dealing with, selling, or exposing, or offering for sale, or having in his possession for the purpose of sale, any quantity of margarine contrary to the provisions of this Act, shall be liable to conviction for an offence against this Act, unless he shows to the satisfaction of the Court before whom he is charged that he purchased the article in question as butter, and with a written warranty or invoice to that effect, that he had no reason to believe at the time when he sold it

## THE ANALYST.

that the article was other than butter, and that he sold it in the same state as when he purchased it, and in such case he shall be discharged from the prosecution, but shall be liable to pay the costs incurred by the prosecutor unless he shall have given due notice to him that he will rely upon the above defence.

[*Margarine imported or manufactured.*] 8. All margarine imported into the United Kingdom of Great Britain and Ireland, and all margarine whether imported or manufactured within the United Kingdom of Great Britain and Ireland, shall, whenever forwarded by any public conveyance, be duly consigned as margarine; and it shall be lawful for any officer of Her Majesty's Customs or Inland Revenue, or any medical officer of health, inspector of nuisances, or police constable, authorised under section thirteen of the Sale of Food and Drugs Act [38 and 39 Vict. c. 63], 1875, to procure samples for analysis if he shall have reason to believe that the provisions of this Act are infringed on this behalf, to examine and take samples from any package, and ascertain, if necessary by submitting the same to be analysed, whether an offence against this Act has been committed.

[*Registration of manufactory.*] 9. Every manufactory of margarine within the United Kingdom of Great Britain and Ireland shall be registered by the owner or occupier thereof with the local authority from time to time in such manner as the Local Government Boards of England and Ireland and the Secretary for Scotland respectively may direct, and every such owner or occupier carrying on such manufacture in a manufactory not duly registered shall be guilty of an offence under this Act.

[*Power to inspectors to take samples without purchase.*] 10. Any officer authorised to take samples under the Sale of Food and Drugs Act, 1875, may, without going through the form of purchase provided by that Act, but otherwise acting in all respects in accordance with the provisions of the said Act as to dealing with samples, take for the purposes of analysis samples of any butter, or substances purporting to be butter, which are exposed for sale, and are not marked Margarine, as provided by this Act; and any such substance not being so marked shall be presumed to be exposed for sale as butter.

[*Appropriation of penalties.*] 11. Any part of any penalty recovered under this Act may, if the Court shall so direct, be paid to the person who proceeds for the same, to reimburse him for the legal costs of obtaining the analysis, and any other reasonable expenses to which the Court shall consider him entitled.

[*Proceedings.*] 12. All proceedings under this Act shall, save as expressly varied by this Act, be the same as prescribed by sections twelve to twenty-eight inclusive of the Sale of Food and Drugs Act, 1875, and all officers employed under that Act are hereby empowered and required to carry out the provisions of this Act.

[*Definition of local authority.*] 13. The expression "local authority" shall mean any local authority authorised to appoint a public analyst under the Sale of Food and Drugs Act, 1875.

### ON REICHERT-MEISSEL'S METHOD OF BUTTER ANALYSIS, AND ITS APPLICATION FOR THE EXAMINATION OF BUTTER AND BUTTER SUBSTITUTES.

BY DR. RUDOLF WOLLNY.

(Continued from page 11.)

Experiments 227 and 228.—5 grms. butter were saponified as usual, and the soap decomposed with 40 c.c. of  $H_2SO_4$  (45 : 10). The distillate required 28.76 c.c. decal-alkali. In a corresponding experiment, 40 c.c. of acid (3 : 10) were used, and the distillate used 29.2 c.c. Still larger quantities were obtained by the use of syrupy phosphoric acid (229-231).



It might, therefore, appear desirable to employ concentrated phosphoric acid, but since the fluid always contains a little alcohol, a quantity of butyric ether must be formed, the larger the greater the concentration of the acid. This was confirmed by a number of experiments (228-232) both with butyric acid solution, and with butter.

As alcohol cannot well be dispensed with in saponification, it becomes imperative to decompose the soap with so small a quantity of sulphuric acid that no free sulphuric acid, but only sodium sulphate with a little bi-sulphate, can be in the solution. In this case, no trace of butyric ether can be formed. Even the concentration 1 : 10 prescribed by the Bavarian chemists, is somewhat too great, and gives rise to the formation of small quantities of ether.

It was further of interest to ascertain the influence of the insoluble fatty acids contained in the retort upon the distillation. It was to be expected that the insoluble acids would have a considerable solvent power upon the butyric acid, and it had to be ascertained how large a quantity of it would be retained by the former.

Experiment 233.—5 grms. butter were saponified, and the soap decomposed as usual. The mixture was heated for one hour in the water-bath, under a reflux condenser. After cooling, the solid layer of fatty acids was removed from the fluid, washed with cold water, and subjected to distillation with 100 c.c. of water, and 40 c.c.  $H_2SO_4$ . 110 c.c. of the distillate required 6.27 c.c. deci-normal solution. The liquor previously decanted from the fatty acids was diluted to 250 c.c., and twice 110 c.c. were distilled off; they required 21.01 and 3.85 c.c., together 24.86 c.c.; total, 31.13 c.c.

Experiment 234.—In an analogous experiment, the decomposed soap was heated for half an hour over the naked flame in an open flask, without boiling. The separated fatty acids, distilled with water and  $H_2SO_4$  as before, used 6.16 c.c. deci-alkali.

Experiment 235.—To the soap of 5 grms. beef fat, 100 c.c. butyric acid solution (requiring 30.6 c.c. deci-solution for neutralisation) were added, and the insoluble fatty acids removed, and distilled with water. 110 c.c. distillate required 2.36 c.c. deci-solution. Hence the fused fatty acids had retained 7.5 per cent. of the total quantity of butyric acid. A similar result was obtained with earthnut oil (237).

Experiments 238 and 239.—2.5 grms. of a butter (of which 5 grms. required for 110 c.c. distillate 28.76 c.c.) were saponified, and treated, in duplicate, as usual, required 15.01 and 15.12 c.c., whilst one half of 5 grms. should have taken 14.38.

Experiments 240 and 241.—5 grms. of a mixture of 50 per cent. of the same butter, and 50 per cent. of earthnut oil, treated as before, used 14.46 and 14.52 c.c., that is but very slightly more than the theory, viz., 14.38, .6 c.c. of the volatile butter acids were retained by the fatty acids of the earthnut oil. The fact that such retention takes place is of great importance. The fatty acids separate from the soap in the form of a soft cheese-like mass, which only becomes completely fused during the latter half of the distillation. The longer the condition of partial fusion continues, the larger the quantity of volatile acids retained.

An experiment has previously been quoted (135) in which only 20.15 c.c., instead of 28.65 c.c. were used in consequence of complete solidification of the fatty acids having taken place. Such large differences of course can only occur in extreme cases, but it cannot be doubted that even much smaller differences of manipulation will be of notable influence upon the result. Even when the operation is apparently conducted in an absolutely uniform manner, a clear condition of the fatty acids is not always obtained in equal times. The low results obtained by Munier (*Zeitsch. f. anal. chem.*, 21, p. 394) are probably due to this cause. It is evidently necessary to remove these inequalities, which can easily be managed by heating the soap under the condenser, by means of a very small flame, until the fatty acids are completely fused, and only then to commence the distillations by increasing the size of the flame.

The quantities and proportions of fat and other materials proposed by Meissl have on the whole been found to be convenient, and it does not appear desirable to take a smaller quantity of fat than 5 grms. Contrary to the statement of Munier I have never found the slightest quantity of sulphuric acid to pass into the distillate.

To test still further the accuracy of the general conclusions arrived at, the same sample of butter was analysed by myself and Dr. A. Sanger, eight times by each according to the method as now perfected, with the following results:—

WOLLNY.		SANGER.	
242	26.40	250	26.40
243	26.40	251	26.35
244	26.45	252	26.40
245	26.45	253	26.40
246	26.40	254	26.40
247	26.35	255	26.40
248	26.40	256	26.40
249	26.40	257	26.40

The figures represent c.c. of deci-normal solution used by 100 c.c. distillate; hence 110 use 29.04, or after subtraction of .28 c.c. for correction, 28.76 c.c.

The time required by the distillation is of great influence upon the result. A butter which required 26.40 c.c. when the distillation lasted half an hour, neutralised 27.2 and 27.5 c.c. when the distillation was so slow as to take 110 and 125 minutes respectively. A difference of a few minutes only does not affect the result.

The following are the sources of error of the Reichert-Meissl method:—

1. Error due to carbonic acid absorbed during saponification (may amount to + 10 per cent.)
2. Error due to formation of butyric ether during saponification (may amount to - 8 per cent.)
3. Error due to formation of ether during distillation (may affect the result - 5 per cent.)
4. Error due to cohesion of fatty acids during distillation (may in extreme cases amount to - 30 per cent.)
5. Error due to shape and size of the vessel in which the distillation is performed, and the time of distillation (may alter the result  $\pm$  5 per cent.).

The following are the details of the method now proposed:—

5 grms. of the clear butter fat are accurately weighed into a 300 c.c. flask (round form, length of neck 7 or 8 c.m., width of neck 2 c.m.), 2 c.c. 50 per cent. soda solution, which must be preserved so that carbonic acid cannot be absorbed, and 10 c.c. of 96 per cent. alcohol, are added, and the mixture is heated under a reflux condenser for 15 minutes in a boiling water bath. The alcohol is then distilled off whilst the flask is heated for at least half an hour; 100 c.c. boiling water are added under due precautions, and the flask heated until the soap is completely dissolved. 40 c.c. of sulphuric acid (25 c.c.  $H_2SO_4$  in 1 litre) and two pieces of pumice of the size of a pea are added, and the flask is at once connected with a condenser by means of a glass tube, 7 c.m. wide, and having, at a distance of 1 c.m. above the cork, a bulb of a diameter of 2.25 c.m. The tube is bent immediately above the bulb upward in an oblique angle, in which direction it extends for 5 c.m., and is then again bent downward, also in oblique angle, and then connected with a condenser by means of an india-rubber tube. The flask is then heated by means of a very small flame, until the insoluble fatty acids are completely fused; 110 c.c. are then distilled off into a graduated flask, the distillation lasting 30 minutes; the distillate is mixed, and 100 c.c. filtered off. These are transferred into a beaker, 1 c.c. phenol-phthalein solution (.5 grms. in 1 litre 50 per cent. alcohol) is added and titrated with deci-normal baryta solution. To the volume of baryta used one-tenth is added, and the figure obtained by blank experiment is subtracted; the latter should not amount to more than .33 c.c.



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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

GENERAL meeting of this Society was held at Burlington House on Wednesday, the 16th February, the President, Mr. A. H. Allen, in the chair.

The minutes of the Annual Meeting were read and confirmed.

Mr. DYER announced that, in pursuance of the resolution passed at the last meeting, the sum of £100 had been invested in Consols.

The PRESIDENT reminded members that this being the beginning of the New Year was a good time for new members to join them, and if they knew any gentlemen who could be an acquisition to the Society they would be glad to welcome them.

The following papers were read and discussed:—

"On a Sample of 'Navy-Green' Paint." By Bertram Blount.

"On the Composition of Milk and Milk-Products." By Dr. Vieth.

"On the Relation between Specific Gravity, Fat, and solids in Milk." By Dr. Vieth.

"Notes on the Estimation of Milk-Sugar in Milk by means of the Polariscopes." By Dr. Vieth.

Mr. H. DROOP RICHMOND exhibited and explained an instrument for calculating milk results, and Mr. STOKES showed a new apparatus for Nesslerising.

The next meeting of the Society will be held at Burlington House on Wednesday, the 14th inst.

## LABORATORY NOTES.

BY ALFRED H. ALLEN, F.C.S., F.I.C.

(Read at the Meeting January 1888.)

*Alumina in Wheat.*—The recent experiments of Yoshida (*Jour. Chem. Soc.*, li., 748) and Young (*ANALYST*, xiii. 5), showing that alumina is a natural constituent of the ash of wheat, have rendered it desirable to see how far their conclusions modify the views hitherto held on this subject.

In the discussion of a paper read by Mr. Wanklyn at the first meeting of this Society, I stated that "in my experience an allowance of 3 milligrammes per 100 grammes of bread, for phosphate of aluminium naturally present, is amply sufficient." This would correspond to about .005 per cent. of aluminium phosphate in the flour. Mr. Jones said his experience confirmed mine absolutely. Dr. Dupré had met with as much as .009 per cent., and Dr. Stevenson agreed with him. Dr. Muter mentioned a case where the bread was made with salt containing alumina had met with nearly .010 per cent. Mr. Wigner was in the habit of allowing .008 for phosphate of aluminium natural to the article.

More recently it has been shown—I think by Dr. Dupré—that the alumina natural to flour bears more or less relation to the proportion of silica present, from which it may be inferred that the alumina exists wholly or chiefly as silicate, and hence is probably of extraneous origin. On the results of these experiments I based the statement in my "Commercial Organic Analysis" (vol. i., page 373), that "it may be taken as a rule that from the amount of alum calculated from the total aluminium of the bread should be subtracted a weight equal to the silica found, when the difference will be approximately the true amount of alum added." Thus if the total aluminium corresponded to .040 per cent. of alum, and the silica was .008 per cent., the corrected proportion of alum would be .032 per cent.

Dr. James Bell considers that his experiments do not indicate any definite relation between the silica and alumina of flour, but his figures show that, broadly speaking, the alumina tends to follow the silica.

The experiments of Yoshida, which appear to have been very carefully made, were conducted on cereals and leguminous plants grown on a soil of volcanic origin, remarkable for containing a large proportion of alumina soluble in hydrochloric acid. Under these circumstances, Yoshida found 2.62 per cent. of mineral matter in the whole wheat, and this mineral matter contained 0.106 per cent. of alumina, equivalent to 0.2536 per cent. of aluminium phosphate. Calculated on the original wheat, this is 0.0066 per cent., or just about what was previously admitted to be normally present.

Mr. Young comes to an exactly similar conclusion, obtaining from Vienna flour of the best quality, containing 0.7 per cent. of ash, 0.0075 per cent. of aluminium phosphate.

All these amounts are so insignificant that they do not materially invalidate the dictum that "flowering plants do not contain aluminium as a normal constituent," though in strict accuracy, and in view of recent experiments, the words "except in extremely minute proportions," should be added. The practical interest attaching to the question exists in the fact that the proportion of alumina found to be naturally present in wheat confirms the justice of making a deduction of 7 or 8 grains of alum per 4 lb. loaf from the amount corresponding to the total aluminium found by analysis.

I may here remind the members that it has been raised as a defence for the presence of alum in flour that alum formed a leading constituent of the composition used for stopping the holes and cracks produced in millstones by wear.

I may call the attention of those interested in the mode of existence of aluminium in bread and flour to a process I devised some years since, by which I obtained very



encouraging results and rendered unnecessary the questionable correction for the aluminium existing as silicate. It consisted in effecting the solution of the starch by malt-extract, destruction of the resultant soluble carbohydrates by fermentation with yeast, acidulation of the liquid by nitric acid, followed by filtration, evaporation of the liquid, ignition of the residue, and precipitation of the aluminium as phosphate in the usual way.

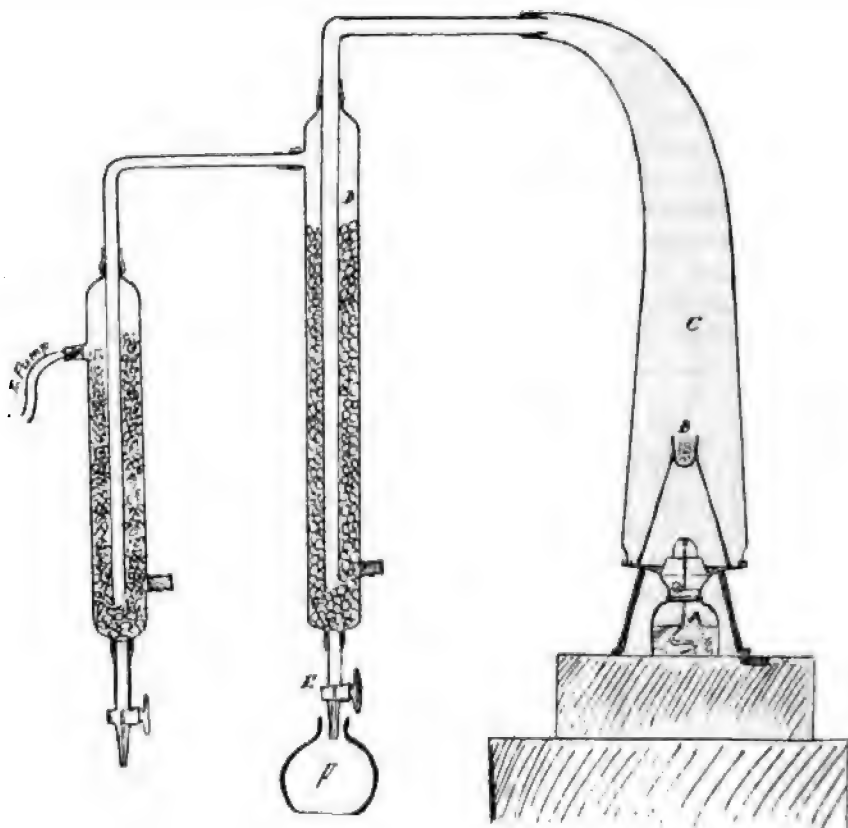
Before leaving the subject, I may add that if the results of the analysis of samples of flour and bread may be regarded as indicating the general character of the articles, adulteration with alum is practically obsolete in the districts for which I act as analyst.

*II. Precipitation of Hop-Bitter by Lead Acetate.*—At a recent meeting of the Society Dr. Johnstone read a paper which was published in the last number of the ANALYST, describing experiments from which he concluded that neither neutral or basic acetate of lead effectually removed the bitter principle from an infusion of hops. As this view was opposed to my own experience, and that of all previous observers, except in a solitary instance mentioned by Dr. Muter, I suggested to Dr. Johnstone that we should both subject to the method the same sample of hops with a view of seeing whether the non-precipitation of the bitter by lead was in any way a consequence of his method of manipulating. In pursuance of this I examined a sample of hops which had been used by Dr. Johnstone in his experiments, and found that the treatment with lead in the manner prescribed by me did not remove the whole of the bitter. On the other hand, I sent a portion of a sample of hops which I had already submitted to the process with satisfactory results to Dr. Johnstone, when curiously enough he failed to precipitate the bitter principle although I had succeeded perfectly. With a second portion of hops from the same source, though not strictly the same sample, he also found the process to fail, in the case of this sample, therefore, there is a direct conflict of evidence, Dr. Johnstone failing to make the process to answer, whereas I had succeeded. But the fact that such an apparently simple operation could fail in the hands of another chemist seemed to me very serious, and I have consequently submitted to the process some eight or ten additional samples of hops. In most instances the process has succeeded fairly well, though when operating on a considerable quantity of hops the residue has usually retained a slight but distinct bitter taste. In one or two instances, however, the bitterness has been more strongly marked, as, indeed, it was in the sample sent me by Dr. Johnstone.

The experiments prove to my mind that precipitation with lead acetate cannot be relied on to remove all the bitter principle of the hop from all infusions containing it, especially when the delicacy of the method is increased by extracting the remaining traces of bitter with chloroform, instead of simply tasting the concentrated filtrate.

*III. Note on the Determination of Sulphur in Oils.*—I had occasion recently to analyse a sample of blast-furnace creosote oil, in which I was desired to determine the proportion of sulphur. The quantity likely to be present was, of course, much smaller than is usually met with in organic substances containing sulphur as an essential constituent, and hence the ordinary processes of determining sulphur, employed in ultimate organic analysis, seemed scarcely applicable, as a considerable quantity of the substance, at least 5 grammes, would be required for the experiment. Oxidation with nitric acid

was for obvious reasons an unsuitable process, and the same remark applied to treatment with dry oxidising agents. Under these circumstances it appeared to me that the best plan would be to burn the oil in a suitable lamp, and estimate the sulphur in the products of combustion, just as sulphur is estimated in coal-gas. I was here met, however, with the difficulty that when burned from a wick the creosote oil produced intolerable quantities of smoke, which could not be controlled and rapidly clogged the wick, whether of asbestos or cotton, thus bringing the experiment to an end. Various experiments in this direction were made until I decided to dilute the creosote oil with alcohol, hoping thus to obtain a better regulation of the combustion. With certain precautions this plan was found to answer, and ultimately the arrangement shown in the sketch was adopted.



A purified methylated spirit was made by distilling the ordinary spirit of commerce from caustic alkali, and this was saturated with solid ammonium carbonate. 5 grammes of creosote oil was then dissolved in 45 grammes of the spirit, and the whole placed in the lamp A, to the top of which the adapter C is fitted air-tight by means of a large bung. The upper end of the adapter is connected with a bent glass-tube passing nearly to the bottom of a vertical Liebig's condenser filled with wet glass marbles and broken glass. The lower end of the condenser is fitted with a glass tap, and the higher tubulure



connected with a water-pump, or other aspirating arrangement by which a continuous steady current of air can be drawn through the apparatus. By preference a second Liebig's condenser, filled with glass beads, is introduced between the first condenser and the pump, but this is not really necessary. The apparatus being arranged, the spirit is ignited, when the combustion proceeds quietly, but the operation has to be conducted very slowly to prevent smoking, and it is desirable to cool the metal part of the lamp by wrapping round it copper gauze, as otherwise the alcohol vapour is liable to kindle with slight explosion, and the lamp is extinguished. The water condensed in D, and which is tapped off at intervals into the flask F, of course contains all the sulphur of the creosote oil in the form of sulphate or sulphite of ammonium. It is treated with lime water, acidulated, and the sulphur precipitated as barium sulphate in the usual way. In this manner the particular sample of creosote oil for which the process was devised was found to contain 0.087 per cent. of sulphur, and on repetition by the same process 0.088 was found.

I have also used the same process for determining the sulphur in rape oil. In this case the sample was mixed with an equal weight of ordinary kerosene, but instead of dissolving the carbonate of ammonium in the liquid it is preferable to suspend it over the flame in a small porcelain crucible. In this way a sample of rape oil was found to contain 0.017 per cent. of sulphur after making a correction for the sulphur in the kerosene oil with which it was mixed, which amounted to 0.000014. A convenient plan for ascertaining how much of the oil has been consumed is to weigh the lamp before and after the experiment.

#### DISCUSSION.

Dr. VIETH said they had heard a very interesting communication from their resident. In a laboratory where there was a variety of work there must always be various results cropping up, and that was just the place to bring those results forward. If that were always done, their meetings would become yet more interesting, and perhaps more members would be induced to contribute papers.

Mr. HEHNER said that with regard to the hop question he had intended to say a few words. He had also taken up the matter, and thought perhaps that a possible explanation of the difference in the results obtained by Dr. Johnstone and Mr. Allen was this—as there was no doubt that those two observers did get different results, and there could be some explanation—perhaps the quantity of acetate of lead used might be the cause, or the precipitate might possibly be more or less soluble in excess of lead solution.

He made an extract from pure hops himself, and added various quantities of lead acetate, removing the excess of lead with sulphuretted hydrogen, and evaporating to dryness, and in all cases he got a residue which was distinctly bitter; also on precipitation with basic acetate one could taste the bitter, but if the liquid was not evaporated to complete dryness, a bitter taste could not be perceived. In no case had he shaken it with chloroform and ether. He thought that the difference greatly arose from the fact that the test had been made rather too delicate, and that it was advisable to avoid evaporation to dryness, or to a very small bulk.

Dr. JOHNSTONE said that he got the bitter out of the beer—not out of the hops. With regard to alumina in flour, a discussion took place in the *Chemical News* some time ago upon the supposed finding of copper in bread. Dr. Edmonds wrote to that journal drawing attention to the blue colour which occurs very often. Other writers made no reply, and then the matter dropped. He had obtained the same results, and

examining the blue colour found it was due to phosphate of iron—vivianite, in fact.

Mr. HEHNER said that whenever he had tested flour for copper he had always found copper.

Mr. ADAMS said that he had made a number of experiments in the hop question, and had never failed to get rid of the bitter when it was due to hops, but perhaps that arose from his not having carried his experiments so far as Mr. Allen had done. He (Mr. Adams) made a decoction, and treated it with basic acetate of lead, threw down the lead finally by sulphuric acid, which was quicker than sulphuretted hydrogen, and then reduced the bulk from half a pint to about half an ounce, and in every case with that quantity he could always taste foreign bitter, while that of the hop had entirely disappeared. He did not know whether that explained Mr. Hehner's results, where he only carried his evaporations to a fluid state, and not dryness. With regard to the detection of bitters, there was only one that failed him, and that was camomile, the bitter of which, like hops, was precipitated by acetate of lead.

Mr. ALLEN said that the direct tasting of the concentrated aqueous liquid was doubtless a less delicate method of recognising a bitter principle than agitating the liquid with ether or chloroform, and tasting the evaporated chloroformic or ethereal solution. It appeared probable that the evaporation of a solution containing more or less free sulphuric acid would tend to decompose any trace of hop-bitter which had remained unprecipitated by lead.\*

#### ON THE COMPOSITION OF MILK AND MILK PRODUCTS.

By DR. P. VIETH., F.C.S., F.I.C.

(Read at the Meeting, February, 1888).

WHAT I wish to bring before you to-night is my annual report on the work done during last year in the laboratory which is under my charge. With regard to the particular circumstances under, and the purposes for which the sample were taken, and also the methods employed for their analysis, I must refer you to former communications of a similar nature. (THE ANALYST, vii., p. 53; viii., p. 33; ix., p. 56; x., p. 67; xi., 66, and xii., p. 39.)

The total number of analyses made during the year 1887 is 20,483, of which 18,611 are analyses of milk.

1,066	"	cream.
661	"	skim milk.
28	"	buttermilk.
13	"	butter and butter-fat.
3	"	cheese.
30	"	medicinal milk preparations.
29	"	water.
42	"	sundry articles.

Besides these analyses, about 80,000 specific gravity determinations have been made during the year.

Of the milk samples analysed 12,663 were taken from the railway churns on their arrival in the dairy, while 2,948 were taken by the inspectors employed by the business from the men, whilst the latter were serving the customers. The monthly averages of all these analyses may be found in the following table.

\* Since the above note was read, I have tried the sulphuric acid treatment on a sample of hops which gave a sensibly bitter extract to chloroform when the lead was removed by sulphuretted hydrogen, and found all trace of bitter had disappeared, even when the concentrated solution was agitated with chloroform, and the evaporated chloroform solution tasted.—A. H. A.



## AVERAGE COMPOSITION OF MILK.

1887.	Samples Taken				
	On Arrival.				On Delivery.
	Specific Gravity.	Fat.	Solids not Fat.	Total Solids.	Total Solids.
ary .....	1.0324	3.77	9.14	12.91	12.92
ary .....	1.0324	3.75	9.15	12.90	12.77
a .....	1.0325	3.69	9.17	12.86	12.77
.....	1.0323	3.62	9.09	12.71	12.64
.....	1.0324	3.75	9.13	12.88	12.87
.....	1.0323	3.71	9.11	12.82	12.78
.....	1.0318	3.66	8.98	12.64	12.60
st .....	1.0315	3.87	8.95	12.82	12.85
nber.....	1.0318	4.12	9.07	13.19	13.15
er .....	1.0324	4.01	9.20	13.21	13.15
nber.....	1.0325	4.01	9.23	13.24	13.13
nber.....	1.0325	3.89	9.21	13.10	13.04
y Average..	1.0322	3.82	9.12	12.94	12.89

From these figures it will be seen that there is a very slight difference between the series of samples, generally in favour of the samples taken on arrival, a difference on a former occasion I have assigned to the difficulty of thoroughly mixing the quantities of milk contained in the railway churns, and to the tendency of cream to rise in the delivery churns. The very minuteness of the differences mentioned sufficiently proves that no serious errors can have been committed in taking the samples. No objection may be raised, that the close agreement of average figures does not exclude differences in individual cases. It is true that such differences may occur, and in fact do occur, but fortunately they are of very rare occurrence.

During last year I have made very extensive series of experiments with a view to investigate into the behaviour of the milk while being delivered to the customers. The principal question to be answered was,—are changes of any importance taking place in the distribution of the fat during the time the milk remains in the delivery churns? Investigation was carried out in January, March and August, and comprises 171 experiments. In each case a sample was taken from the well-mixed milk contained in the delivery churn before the latter left the yard, and a second sample of the small quantity of milk left in the churn on return of the man from his round. I do not weary of harassing you with the results of 342 analyses, but shall confine myself to stating that in 2 cases out of 171, or 1.2 per cent., the samples of milk returned from the churn contained a plus of more than .3 per cent. of fat, viz., .32 and .34, in 8 cases, or 4.7 per cent., a plus of from .2 to .3 per cent., and in 20 cases, or 11.7 per cent., a plus

of from .1 to .2 per cent. of fat. Here then we cannot speak of serious changes in the distribution of the fat, and it is very fortunate that this appears to be the rule under the conditions under which the business with which I am connected, and similar businesses are working. That exceptions do occur, I have brought under your notice on several occasions, when laying before you cases observed by myself of abnormally quick rising of cream in milk whilst being delivered to the customers. (THE ANALYST, viii., p. 2; ix., p. 57.)

Samples of cream as supplied to customers were regularly taken before the cream was sent out, as well as from the men on the rounds. The average amounts of fat found in the two series of samples are contained in the following table.

AVERAGE AMOUNT OF FAT IN CREAM.

1887.	Samples Taken	
	Before sent out.	On delivery.
January ... ..	46.1	45.5
February ... ..	43.7	43.5
March ... ..	43.2	43.4
April ... ..	43.3	43.3
May ... ..	45.6	45.3
June ... ..	43.3	43.1
July ... ..	42.6	41.7
August ... ..	40.6	41.2
September ... ..	40.6	41.1
October ... ..	41.9	42.4
November ... ..	43.2	43.5
December ... ..	44.2	44.8
Yearly Average ... ..	43.2	43.2

The composition of 51 samples of clotted cream was as follows:—

	Limits.	Average.
Water .. ..	31.16 to 44.16 ..	36.94 per cent.
Fat .. ..	46.92 „ 62.00 ..	55.51 „
Solids-not-fat ..	5.26 „ 11.41 ..	7.55 „
Ash .. ..	.46 „ .85 ..	.58 „

In skim milk samples the fat was usually found to amount to from .15 to .40 per cent., and in very exceptional cases only above .40 per cent. was present.

Ten samples of butter were analysed with the following results:—

	Limits.	Average.
Fat .. ..	82.57 to 88.34 ..	85.14 per cent.
Water .. ..	9.54 „ 14.39 ..	12.93 „
Proteids, etc. ..	.44 „ 1.26 ..	.90 „
Ash, including NaCl ..	.09 „ 3.15 ..	1.03 „
Insoluble fatty acids	87.40 „ 88.86 ..	88.08 „



A sample of whey butter contained :—

Fat 86.35 per cent. ;	Proteids, etc., 70 per cent. ;
Water 10.58 per cent. ;	Ash, 2.37 per cent.

In two samples of butter-fat, which had been kept for a year exposed to the action of air and light, and had attained a waxy appearance, the insoluble fatty acids were reduced in the one case from 88.33 to 85.97, and in the other from 87.61 to 84.41 per cent.

There were also some examinations of other fats made with the following results :—

Beef Tallow, melting point 49° C. ; insol. fatty acids, 95.23 per cent.	
Mutton Tallow       "       49° C.,       "       "       94.82       "	
Lard                 "       41° C.,       "       "       95.04       "	
Olive Oil . . . . . "       "       95.18       "	

A sample of Cheddar cheese which was sent to me analysed as follows :—

Water, 38.31 per cent. ; Fat, 29.13 per cent. ; Casein, etc., 29.47 per cent. ; Ash, 3.09 per cent. ; Cl, 37 per cent.

So far the cheese was of quite normal composition, but when I came to analyse a larger quantity of fat extracted from the cheese I found the insoluble fatty acids to amount to 92.76 per cent., and the quantity of deci-normal potash required to neutralise the distillate from 2.5 grms. to be .9 c.c. What I had under my hands was an artificial fat cheese, which, I must admit, was very well made.

## ON THE RELATION BETWEEN SPECIFIC GRAVITY, FAT, AND SOLIDS IN MILK.

By DR. P. VIETH, F.C.S., F.I.C.

(Read at the Meeting, February, 1888).

The relation between specific gravity, fat, and solids in milk has been before the Society a very short time ago. As the subject is however, one of great interest and the very greatest importance, I venture to make a few observations on it to-night.

At the December meeting Mr. Hehner brought before us an extensive series of experiments showing the percentages of fat extracted from milk dried on paper coils, and calculated from his new formula. The agreement is very close indeed, and the calculation leaves nothing to be desired. In the discussion on Mr. Hehner's paper I took occasion to remark that the experiments at the same time bore out a fact to which I have repeatedly drawn attention, viz., that the differences between the percentages of fat extracted if the coil process is employed, or calculated from Hehner's new formula, and fat extracted if the plaster process is used, or calculated from Fleischmann's formula, are smallest with skim milks, and increase with the increasing percentage of fat ; or, in other words, that the differences are small where great difficulties for the complete extraction of fat exist, and comparatively large where no such difficulties are present.

Thus, in Mr. Hehner's series of analyses the following differences occur :—

5 samples with	.1 to 1.0 per cent. fat ;	average difference,	— .02 per cent.
3	"   "   2.1   "   3.0   "   "   "   "   "		— .18   "
14	"   "   3.1   "   4.0   "   "   "   "   "		— .23   "
10	"   "   4.1   "   5.0   "   "   "   "   "		— .30   "
7	"   "   5.1   "   6.0   "   "   "   "   "		— .31   "
1	"   "       6.20   "   "   "   "   "   "		— .24   "
1	"   "       11.63   "   "   "   "   "   "		— .41   "
1	"   "       24.06   "   "   "   "   "   "		— .65   "

If you take the trouble to look into THE ANALYST, xii., p. 63, you will see that I have found similar results two years ago.

No explanation has as yet been offered for this very curious fact, which is the reason that I have not adopted the coil process, although I admit that it is rather more convenient and simple than the plaster process, especially if worked in the way suggested by Mr. Hehner.

Results obtained by employing the plaster process, which I am still in the habit of using, must be compared with figures calculated from Fleischmann's formula. I thought it interesting to compile a greater number of such figures than are usually obtainable. Looking through my laboratory journals since 1881, I extracted 694 analyses, which allow to compare figures for fat extracted and fat calculated. Among these analyses there are a great number which have been made with the utmost care and in duplicate, while in other cases there was neither occasion nor opportunity for making the analyses in duplicate. I give here the results of 628 analyses in a tabulated form.

Number of Samples.	Fat.	Solids not Fat.	Specific Gravity.	Difference.	
				Limits.	Average.
MIXED MILKS.					
141	0.1 to 0.5	8.8 to 10.0	1.0345 to 1.0385	-.2 to +.2	+.04
16	0.6 „ 1.0	5.3 „ 9.8	1.0200 „ 1.0370	-.1 „ +.2	+.03
11	1.1 „ 1.5	6.7 „ 9.6	1.0245 „ 1.0370	-.1 „ +.1	-.02
11	1.6 „ 2.0	5.8 „ 9.7	1.0210 „ 1.0360	-.1 „ +.2	+.02
21	2.1 „ 2.5	5.1 „ 9.4	1.0180 „ 1.0345	-.2 „ +.2	+.03
43	2.6 „ 3.0	6.7 „ 9.7	1.0230 „ 1.0350	-.1 „ +.2	+.03
124	3.1 „ 3.5	7.5 „ 10.0	1.0265 „ 1.0360	-.2 „ +.2	+.01
95	3.6 „ 4.0	8.0 „ 9.7	1.0280 „ 1.0350	-.2 „ +.2	+.01
38	4.1 „ 4.5	8.1 „ 10.1	1.0300 „ 1.0350	-.2 „ +.2	+.05
14	4.6 „ 5.0	8.9 „ 9.6	1.0305 „ 1.0350	-.1 „ +.2	+.01
16	5.1 „ 10.0	8.6 „ 9.8	1.0255 „ 1.0335	-.2 „ +.1	-.02
MILK OF INDIVIDUAL COWS.					
33	2.0 „ 7.5	6.4 „ 10.8	1.0205 „ 1.0350	-.2 „ +.2	+.05
BUTTERMILK.					
65	.4 „ 3.1	7.3 „ 9.9	1.0260 „ 1.0355	-.2 „ +.2	+.02

There are, first of all, 530 samples of milk of very widely varying degrees of richness: poor skim milks, half-skimmed milks, milks with an average amount of fat, rich milks, and mixtures of milk and cream. Included are a number of samples to which water had been added. The differences between fat found and fat calculated range from - .2 to + .2 and average + .02.

All these samples refer to mixed yields of several, in most instances of a good many, cows, and the question arises, whether in the case of milks of individual cows the calculation is applicable with the same good result. This question is answered in the affirmative by the thirty-three analyses which follow next in the table, showing the same range of differences as the previous samples, viz., from - .2 to + .2 and an average difference of + .05.



We now come to sixty-five analyses of buttermilk, which were either perfectly sweet or at least of a degree of acidity, and on the whole in a condition allowing the specific gravity to be determined with sufficient accuracy. Here again we have differences ranging from  $-.2$  to  $+.2$  and averaging  $+.02$ .

The possibility of applying formulæ of the kind in question is, of course, based upon the assumption that the specific gravity of the milk fat on the one hand and the non-fatty solids on the other are practically constant figures. We know that the specific gravity of pure butter fat is  $.93$ , and Fleischmann, when working out his formula, has found that the specific gravity of non-fatty solids is  $1.6$ ; but what the specific gravities of the several solid component parts are, as long as they, in conjunction with water, constitute milk, we know not. There can, however, be no doubt that proteids, milk-sugar, and the mineral salts, forming the non-fatty solids, have different specific gravities, and I believe we may reasonably assume that the specific gravities decline in the order, ash, sugar, proteids. If the specific gravity of the non-fatty solids is always a constant, or nearly constant, quantity, we must admit that the relative proportion in which the three constituents are present is also constant, or nearly so. Roughly speaking one half of the non-fatty solids is sugar, the sixth part of the other half ash, and the remainder proteids; or, to put it in other words, ash, proteids, and sugar, are present in the proportion of  $1 : 5 : 6$ . As long as this is the case, formulæ like Fleischmann's and Hehner's can be applied with satisfactory results.

As soon, however, as this relative proportion is seriously disturbed, the application of the formulæ with anything like success becomes impossible. The following figures prove that beyond doubt. In artificial human milk, as manufactured under my superintendence, the relative proportion of ash, proteids, and sugar is  $1 : 5 : 10$ ; in seventeen analyses the differences, when the formula was applied, ranged from  $-.4$  to  $0.0$ , and averaged  $-.21$ . In special milk food, another medicinal preparation for infants, the relative proportion is  $1 : 3 : 10$ ; and in eleven analyses differences from  $-.7$  to  $-.2$  average  $-.42$ , were found. In mare's milk, which I had the opportunity of analysing some years ago, the proportion in question is  $1 : 6 : 23$ ; in thirty-one analyses the differences varied from  $-.6$  to  $0.0$ , and averaged  $-.25$ .

Lastly, I applied the formula to analyses of condensed milks, diluted or dissolved in proportion as prescribed, when I got the following results:—

1 sample milk powder .. .. .	difference	$-.1$	per cent.
2 samples milk, condensed without sugar ..	"	$0.0, -.1$	"
1 sample " " with sugar ..	"	$-.4$	"
3 samples condensed mare's milk ..	"	$-.3, -.1, -.5$	"

In conclusion, I venture to express my belief that the figures contained in what precedes clearly prove how very useful and widely applicable Fleischmann's as well as Hehner's formula are, for both formulæ will give equally satisfactory results if applied in their proper sphere. At the same time my figures indicate where the application of the formulæ has to stop.

*(Conclusion of the Society's Proceedings.)*

## THE PRECIPITATION OF HOP-BITTERS BY MEANS OF NEUTRAL OR SUBACETATE OF LEAD.

BY HENRY J. HARDY, F.C.S.

FROM time to time various methods have been proposed and used for the separation and recognition of the bitter principle of hops from that of other bitters used as hop-substitutes. Some of these methods are old, while others are of recent date. It matters little whether the process is old or new, as they all resemble each other very much, being no doubt modifications of one another.

The principle of each process is that the bitter of hops is said to be completely precipitated by lead acetate.

A short time ago doubt was thrown upon this statement, and this led me to go into the matter. I have made a number of experiments with pure hops, using for each experiment an infusion made from seven to eight grammes of hops, which would approximately correspond to the amount present in a litre of an average bitter beer. I also used beer brewed in the laboratory from pure malt and hops. I based my experiments upon the method of Allen published in *THE ANALYST*, xii., 134, and also upon that of Enders. In no case was I able to remove the hop-bitter by means of neutral or subacetate of lead, therefore I say without any doubt whatever that lead acetate is altogether useless in precipitating the bitter of hops as it does not do so completely, the most bitter principles remaining in solution.

These results confirm those obtained by Hayduck, who found\* that the bitter of hops is due to the presence of three feeble resinous acids which vary in their bitter intensity. One of these, which is moderately bitter, is precipitated by lead acetate, whilst the remaining two, which are intensely bitter, are not.

It is thus shown that neutral or subacetate of lead does not precipitate completely the natural bitter of hops, and therefore any process based upon this assumption is utterly worthless.

## NOTE ON A TEST FOR "HYDRONAPHTHOL."

By ALFRED L. BEEBE, ASSISTANT CHEMIST, NEW YORK CITY HEALTH DEPARTMENT.

"HYDRONAPHTHOL," so-called, has lately come into considerable prominence as an effective antiseptic for use in the preservation of various food products. A reliable method for its detection is therefore desirable. So far as the writer is aware, no distinctive test has as yet been given for the detection of this substance, when present in minute quantities, and the results of some experiments in this direction may therefore prove of interest.

"Hydronaphthol," as is well known, is really a trade name for  $\beta$  naphthol. It is sparingly soluble in cold, much more readily in hot water, and is easily extracted from its water solution by shaking with ether. The etherial extract, evaporated to dryness and taken up with hot water, or the water solution direct, in absence of interfering substances, made slightly alkaline with ammonia, cooled, and slightly acidified with dilute nitric acid, gives, on addition of a drop of fuming nitric acid or a nitrite, a beautiful rose color, analogous to that developed in the test for nitrites in water.

\* *Bied. Centr.*, 1887, 694-695.



In making the test care should be taken that the ammonia and nitric acid are actively added in slight excess only, and that the nitric acid used is so dilute as to require no heating of the solution by its combination with the excess of ammonia present. If these precautions are not observed, a dirty salmon colour is apt to be developed by the action of the fuming nitric acid, and the distinctive character of the test thereby destroyed.

The reaction is one of extreme delicacy, one part in ten thousand of "hydronaphthol" being readily detected. Experiments are now being made by the writer, to determine the limit of delicacy of the test, and also to arrive at a practical method for detecting "hydronaphthol" from food products to which it may have been added, in a solution suitable for the application of the test outlined above. The results of these experiments, if successful, will be published in due course.

#### DO CEREALS CONTAIN SUGAR.\*

BY PROF. DR. ALEX. V. ASSBÖTH.

My paper on the determination of starch (see ANALYST, July, 1887) I came to the conclusion that cereals do not contain any sugar. Baryta water does not precipitate sugar in presence of alcohol, and still my full analyses of various cereals came close up to 100 per cent., leaving, therefore, no room for any sugar. As, however, Lish pretends that, by following the details of my process, the sugar cannot be detected, and many chemists of repute have found sugar, I once more investigated the matter so as to prove myself in the right.

TABLE, SHOWING THE AMOUNT OF SUGAR FOUND IN NON-GERMINATED BARLEY AND WHEAT BY SEVERAL AUTHORITIES (KÜHNEMANN).

("0" means not found; "—" means not tried for.)

		BARLEY.		WHEAT.	
		Sugar. Per cent.	Dextrin. Per cent.	Sugar. Per cent.	Dextrin. Per cent.
Thomson (1817)	..	4	—	—	—
Polson	..	—	4.8	—	5.3
Groust (1817)	..	5	4	—	—
Peligot	..	0	—	—	—
Boussingault	..	—	—	—	7.2
Saussure	..	—	—	2.4	3.4
Mulder	(1858)	0	—	—	—
Oudemans					
Mitscherlich	..	0	—	—	—
Hermstaedt	..	4.7	4.5	—	—
Einhof (1838)	..	5.2	4.6	—	—
W. Stein (1860)	..	0	6.5	—	—
Pillitz (1872)	..	2.71	1.96	1.60	1.76

According to Oudemans, analysts have now and then found sugar, because they extracted the flour with water, which caused the dextrin, and probably some of the starch, to invert. If flour is extracted with absolute alcohol, and the solution evaporated to dryness, a residue is obtained which, after inversion with strong acetic acid, reduces the copper solution, and yields about .2 per cent. of sugar; but even these small traces are not really present as such in the original flour. Most likely they are derived from a little dextrin.

\* Chem. Zeit., 2 and 4, 1888.

as this substance is not quite insoluble in alcohol, and flour probably contains small quantities of copper-reducing substances which need not necessarily be sugar.

Krocker extracts the flour with limewater and filters. The filtrate is freed from albuminoids and excess of lime by a current of carbonic acid. Sugar could not be detected in the filtrate.

Schlösing estimates sugar as follows:—10 to 15 grammes of finely ground substance are repeatedly rubbed in a mortar with small quantities of water, and finally exhausted in a displacement apparatus. The filtrate is divided into three equal parts. Sugar is estimated by Fehling's solution in one half of one of these parts. The other portion is used for determining the dextrin.

Koenig modifies this process. He evaporates the watery solution and then extracts with rectified spirits of wine. The alcohol is distilled off and the residue dissolved in a definite volume of water. If much colouring matter is present some animal charcoal must be used. The filtrate is divided into two equal parts and the glucose estimated, best by the well-known mercury solution. The other part is heated with a few drops of sulphuric acid for two hours in the water-bath, and then treated like the first. Any increase in sugar is calculated to cane-sugar.

J. Bell gives the following method:—10 grammes of finely powdered material are extracted with alcohol of 70 per cent. The alcoholic solution is evaporated, and the residue inverted with 5 c.c. of normal sulphuric acid. The sugar is then estimated by one of the well-known processes. To practically ascertain whether these processes give reliable results, and to prove my case, I have made the following experiments:—

1. 10 grms. of maize-flour were gelatinised with 50 c.c. of water, and, whilst still warm, mixed with 200 c.c. of 80 per cent. alcohol. After cooling, the liquid was filtered and evaporated to dryness. I took up the residue with a little water and tested a little of it with Fehling. Notwithstanding the fluid gave a copious precipitate with baryta water and proof spirit, I could scarcely get any reduction of the copper solution.

2. About 15 grammes of maize-flour were extracted in the cold with 200 c.c. of 80 per cent. alcohol; the latter evaporated and the residue dissolved in water. This solution (which but sparingly reduced Fehling) was mixed with lime water and boiled. The excess of lime and other impurities were removed by carbonic acid and the filtrate evaporated to dryness. The residue was again treated with rectified spirits of wine, when an insoluble yellow powder remained undissolved. This was impure dextrin. The filtrate had a brownish colour, and left, after evaporation, a brownish sticky mass, readily soluble in water. This solution is precipitated by baryta water in presence of alcohol. The substance is, no doubt, erythrodextrin. With iodine it gives a red colour, and it does not reduce Fehling until it has been boiled with hydrochloric acid.

3. Experiments with wheat and peas gave similar results.

4. 20 grms. of maize were extracted according to Schlösing's process. The solution strongly reduced the alkaline copper solution.

I now come to the following conclusions:—*The cereals contain no sugar, neither glucose nor saccharose.* Analysts who have apparently found it have made use of wrong processes.

Schlösing's process is wrong, because the erythrodextrin, and most likely some of



o starch, yield a little glucose by mere treatment with water. Koenig's modification also wrong, as my experiments plainly prove cereals contain a gummy-like substance, soluble in alcohol, which yields glucose on boiling with acids. The process of J. Bell and Rasenach also gives inaccurate results, as dextrin is perceptibly soluble in rectified spirits of wine, and the subsequent boiling with acid will make this into glucose. There is also the dextrin-like substance which I have found in all cereals, and which yields glucose on boiling with acids.

As dextrin and erythrodextrin slightly reduce copper solution on prolonged boiling, it is not to be wondered at I sometimes got, in my analyses, *slight* reductions which ought to have taken place.

### THE ACTION OF ALCOHOL ON BUTTER FAT.

By PROF. C. B. COCHRAN, WEST CHESTER, PA., U.S.A.

In order to test artificial butter for added butyrates it has been recommended that the suspected sample be treated with alcohol, which will dissolve any artificial tributyrate. If the undissolved fat then be tested by Reichert's method, we shall find the per cent. of volatile fat acids much less than in the original fat, in case any artificially prepared butyrates have been used.

If this plan of examining suspected butters is ever to be called into practical use, it is, in my opinion, highly desirable, if not absolutely necessary, that we have some definite knowledge of the action of alcohol upon genuine butter fat, under certain fixed conditions, such as could easily be adopted when examining suspected samples.

The following experiments were made for the purpose of determining what changes, if any, were produced in butter by treating it with ethyl alcohol. The alcohol used gave specific gravity corresponding to 90 per cent.  $C_2H_5O$ . The amount used was 10 c.c. of alcohol to each gramme of butter fat.

C.c.  $\frac{N}{10}$  KHO required for distillate of 50 c.c.  
from  $2\frac{1}{2}$  grms.  
(REICHERT'S METHOD.)

Sample.	Fat dissolved by 10 c.c.	Temperature.	Butter fat.	Dissolved fat.	Undissolved fat.
1.	0.1083	78°F.	13 c.c.	24 c.c.	
2.	0.1086	78°F.		25 $\frac{3}{10}$ c.c.	9 $\frac{1}{2}$ c.c.
3.	0.130	76°F.	{ 11 c.c. 10 $\frac{1}{2}$ c.c.	19 $\frac{1}{2}$ c.c.	9 $\frac{1}{10}$ c.c.
4.	0.120	75°F.	15 $\frac{3}{10}$ c.c.	27 $\frac{3}{10}$ c.c.	13 $\frac{1}{10}$ c.c.
5.	0.126		12 $\frac{9}{10}$ c.c.	28 $\frac{4}{10}$ c.c.	{ 12 $\frac{3}{10}$ c.c. 12 $\frac{4}{10}$ c.c.

Sample No. 4 giving in one case only 10  $\frac{1}{2}$  c.c., and 11 c.c.  $\frac{N}{10}$  KHO in another, to neutralise the distillate from  $2\frac{1}{2}$  grms. of fat was more than 10 months old. In a third set of this same butter known to be genuine 10  $\frac{1}{2}$  c.c.  $\frac{N}{10}$  KHO were required. This butter was at the time in a state of good preservation, and was perfectly palatable.

Hehner and Angell, in their work on butter analysis, found that the insoluble fat acids of butter increased somewhat with age. This being the case we might expect some decrease in the per cent. of soluble fatty acids, and this result, therefore, seems to be in accord with the report of Hehner and Angell.

Three other samples of butter tested in a similar way, but no attention paid to the condition of temperature, gave results as follows:—

## PER CENT. OF VOLATILE FAT ACIDS IN

Sample.	Butter fat.	Undissolved fat.	Dissolved fat.
1.	$6\frac{3}{10}$ per cent.	$5\frac{23}{100}$ per cent.	$10\frac{15}{100}$ per cent.
2.	$5\frac{96}{100}$ "	$3\frac{1}{10}$ "	$14\frac{43}{100}$ "
3.	$5\frac{39}{100}$ "	$2\frac{71}{100}$ "	$11\frac{87}{100}$ "

The per-cent. of volatile fat acids in the above table was determined in each case by the process of repeated distillations and calculated as butyric acid.

Experiments made with wood alcohol show similar results. I here give results of two such experiments, the proportion of alcohol to fat being 10 c.c. to 1 grm.

C.c.  $\frac{N}{10}$  KHO required to neutralise 50 c.c.  
distillate from  $2\frac{1}{2}$  grms.

Sample.	Fat dissolved by 10 c.c.	Temp.	Butter fat.	Undissolved fat.	Dissolved fat.
1.	225	75°F.	$15\frac{6}{10}$	$9\frac{6}{10}$	$23\frac{1}{3}$
2.	218	75°F.	$10\frac{1}{2}$	$1\frac{24}{100}$	$22\frac{9}{19}$

The second sample of butter was the same as mentioned above, and was ten months old.

From the above work it is very evident that the portion of fat dissolved by methyl or ethyl alcohol is rich in volatile fat acids. We conclude, therefore, that while the glycerides of the volatile fat acids are present in butter in such a condition as not to be readily dissolved by alcohol, yet a part of them yields to the solvent action of this reagent more readily than some of the other fats of the butter. This excess of volatile fat acids in the fat dissolved by alcohol is not due simply to the solvent action of alcohol upon any free fat acid which may be present, as my method of experimenting eliminated this possibility.

If one suspects a butter of being adulterated with an artificial butyrate, and attempts to dissolve such an adulterant by the use of alcohol, it seems necessary to use an alcohol of known strength and to have a known quantitative relation between the alcohol and butter fat employed. If the fat undissolved by alcohol be then examined he must expect to find the quantity of volatile fat acids diminished somewhat even in genuine butter.

Using the method I have adopted I should judge that the undissolved fat of a genuine butter ought to require 9 c.c. of  $\frac{N}{10}$  KHO to neutralise the usual distillate from  $2\frac{1}{2}$  grammes. If less than nine are required there would be reason to suspect adulteration.

I am inclined to think that more information would be gained by applying Reichert's method to the fat undissolved by alcohol than by applying it to the original fat if we adopt 9 c.c.  $\frac{N}{10}$  KHO as the amount of alkali required to neutralise the distillate from  $2\frac{1}{2}$  grammes of fat strictly following Reichert's method. This would in most cases show at once the adulteration even in case of an added butyrate; when the same method applied to the suspected butter would not detect this.

Hübl's iodine test also shows that the composition of fat dissolved by alcohol differs from the portion undissolved.

In the following tests the fat was dissolved in wood alcohol of specific gravity .817 at 60° F. The proportion of alcohol used was 10 c.c. to one gramme of fat.



Sample.	Fat dissolved by 10 c.c.	Temp.	Iodine No. of fat.	Iodine No. of dissolved fat	Iodine No. of Undissolved fat.
1.	·225 grm.	75° F.	37	$33\frac{7}{10}$	$38\frac{3}{10}$
2.				$34\frac{6}{10}$	$37\frac{1}{2}$
3.	·218 grm.		$45\frac{3}{10}$	$43\frac{1}{10}$	$45\frac{8}{10}$

Below are two determinations of the iodine numbers when ethyl alcohol 90 per cent.  $C_2H_5O$  was used to dissolve the fat.

Sample	Fat dissolved by 10 c.c.	Temp.	Iodine No. of fat.	Iodine No. of dissolved fat.	Iodine No. of Undissolved fat.
1.	·126 grm.		$40\frac{4}{10}$	$37\frac{1}{10}$	$42\frac{2}{10}$
2.	·1197 „	82° F.	$41\frac{7}{10}$	$36\frac{8}{10}$	$42\frac{2}{10}$

The fat dissolved by alcohol has a very low melting point, remaining liquid at all ordinary temperatures, while the undissolved portion has a melting point higher than pure butter fat. In one case the undissolved portion had a melting point of 106° F.

An attempt was made to determine the character of the fat acid condensed in condenser during distillation with the following results :—

The fat acid collected in condenser was dissolved in neutral alcohol, and titrated with  $\frac{N}{10}$  baric hydrate; this formed a heavy white precipitate.

Weight of Ba salt thus formed = 0·1325 grm.

Weight of  $BaSO_4$  produced from this salt = 0·063 grm.

Weight of  $BaSO_4$  which would correspond to 0·1325 grm. of  $Ba(C_{10}H_{19}O_2)_2$  = 0·0644.

The melting point of the fat acid collected in condenser was twice determined, and in one case was found to be 80° F., and in the second case 86° F. The last melting point corresponds to that of capric acid. From the above results I judge that the fat acid collected in condenser is composed mostly of capric acid.

Before concluding this article I wish to say that in my opinion the standard of  $12\frac{1}{2}$  c.c.  $\frac{N}{10}$  KHO as frequently adopted for Reichert's method will need some modification. While fresh butter will give results as high as this standard, or exceeding it, I believe that in case of butters that have been kept for some time we may obtain results short of this. At least this has been my experience with butter made at my home in the fall, and kept over winter, although the butter was still very palatable.

#### AN IMPROVED FORM OF GAS APPARATUS.

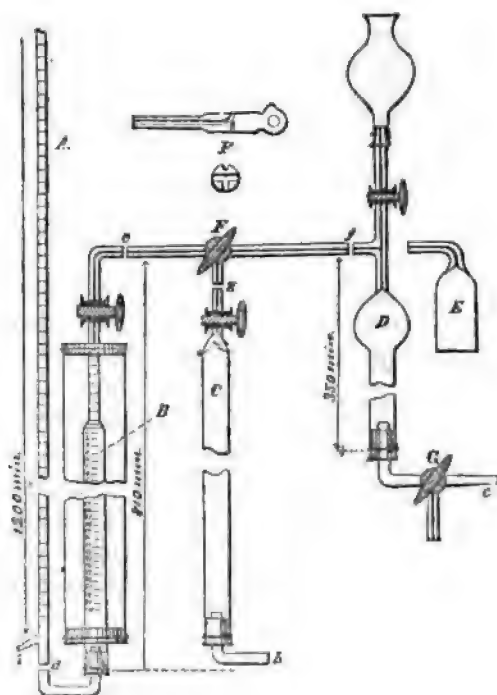
By J. T. WILLARD.\*

THE description of an improved form of Elliott's apparatus for gas analysis, by J. B. Mackintosh, recalls one devised by the writer. It was constructed for use in the chemical laboratory of the State Agricultural College of Kansas, and was used in an examination of the natural gases of that State by Prof. G. H. Failyer. As it embodies some advantages not combined in any other apparatus that has come under the notice of the writer, a description of it may not be amiss. It is essentially a combination of Elliott's apparatus and Frankland's apparatus for the analysis of gases incidental to water analysis,† with important modifications and additions. The accompanying cut will make its construction clear.

\* American Chemical Journal.

† Journal Chem. Society 21, 109.

*A* is a pressure tube graduated in millimetres. *B* is the measuring tube holding about 120 c.c., 100 c.c. of which is graduated to tenths, beginning with the stopcock *F*. Its upper part is narrow, thus admitting accurate measurement of small amounts of gas as well as large. It is enclosed in a water jacket which must be provided with some means of securing a uniform temperature throughout. *C* is the explosion tube and is ungraduated. *D* is the absorption tube surmounted by a funnel for the introduction of reagents. *E* is a laboratory vessel of the ordinary kind, which may be attached instead of *D* if desired. It is obvious that any form of absorption pipette may be attached at *f*. *B*, *C* and *D* are connected by the stopcock *F*, a four-way cock shown in section above. By means of this cock, the others being suitably arranged, either tube may be



put in connection with either of the others or with the external air, without disturbing the other tube or tubes. *G* is an ordinary three-way cock. Reservoirs not shown in the cut are attached to the tubes by means of rubber tubing at *a*, *b* and *c*. The ends of the tubes are closed by rubber stoppers, and the several parts are connected by heavy rubber connectors at *d*, *e*, *f*, and *g*. The ends of the capillary tubes are ground squarely at these joints so that they come together perfectly. The apparatus is firmly attached to a suitable support, such as any one with a little ingenuity may devise. It is essential that *A* and *B* be so supported that their relative position shall remain unaltered. The apparatus was designed for use with mercury, but water may be used. With water the pressure tube would be unnecessary.



Concerning the mode of operating the apparatus but little need be said. It is most convenient to use Frankland's method of measuring, in which the gas operated upon is always brought to the same volume, or to an aliquot part of the original volume, by adjusting the pressure of the mercury in *A*. Points on the pressure tube corresponding to a number of convenient volumes in the measuring tube must be previously determined with care. The tensions exerted by varying amounts of gas brought to the same volume will be proportional to the amounts of gas present. If the gas is brought to an aliquot part of the original volume, the tension found may be reduced to that corresponding to the original volume by a very simple calculation.

The explosion of the gases is performed under reduced pressure according to the principles developed by Thomas. This method of explosion is very advantageous and may be used with this apparatus, even if water is used in the tubes *B* and *D*, by filling *C* with mercury. On lowering the reservoir connected with *C* any desired degree of rarefaction may be produced. It is convenient to have a rough scale back of *C* and extending below, for use in measuring the pressure under which the gas is confined during explosion.

The fourth way in the stopcock *F* is essential for the discharge of the reagents employed in *D*; it is also used for the introduction of the gases. If it is desired to preserve a portion of the gas in *D* while another portion is being measured and exploded in *B* and *C*, it is necessary to close this external opening of *F*. This may be done simply and perfectly by filling the way with water or mercury from one of the tubes, and then slipping a short piece of rubber tubing filled with water over the end of the stopcock and closing it with a clamp.

I think no other details of manipulation need be entered into, as they are similar to those already described for other apparatus, or can be readily worked out by the operator.

The apparatus may be used for certain of the purposes to which the nitrometer has been put, such as the valuation of bleaching powder by hydrogen peroxide and vice versa.

The apparatus described was made in most excellent manner by Mr. Emil Greiner, New York.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

REACTION OF COTTON-SEED OIL. M. LABICHE. *L'Union Ph.*—Treated with subacetate of lead and caustic alkali, cotton-seed oil gives, almost immediately, an orange-red reaction. This is peculiar to this oil, for almond, castor, olive, poppy, rape, and cod-liver oils give a milky mixture, which is also the case with butter when thus treated. The author mixes equal parts of the oil and a saturated solution of neutral acetate of lead, and adds ammonia, stirring briskly. The acetate decomposes, and the nascent oxide reacts upon the oil. Then the red colour appears. After standing, the surface turns orange-red and the lower portion becomes grumous. If 20 per cent. of cotton-seed oil be present, the colouration appears at once; lesser quantities show on the surface after the mixture has remained standing for a time.

W. H. D.

## REVIEW.

FOODS: THEIR COMPOSITION AND ANALYSIS. A MANUAL FOR THE USE OF ANALYTICAL CHEMISTS AND OTHERS. WITH AN INTRODUCTORY ESSAY ON THE HISTORY OF ADULTERATION. BY ALEXANDER WYNTER BLYTH, M.R.C.S., F.C.S., with numerous tables and illustrations. Third edition. (London: Charles Griffin and Co.)

THE third edition of Mr. Blyth's standard work is now placed before the public, and will, doubtless, be received with the same favour that was universally extended to the former editions. Mr. Blyth fully recognises the necessity of keeping up with the times, and accordingly we find a mention of nearly everything in the shape of improvements, or supposed improvements, which has appeared in our columns and elsewhere, on the subject of the analysis of food. At the same time, certain processes that have been exposed to the furnace of experience and have not issued therefrom unsullied are judiciously omitted in the present book. To attempt anything in the shape of a criticism of so well-established a work would be simply an act of supererogation, because the few defects that lurk among its many merits have long ago been discussed, and the author, doubtless, has his own good reasons for retaining certain points which may not receive universal approbation. The fact, however, remains, that Mr Blyth's book is still *facile princeps* among works in the English language devoted to the subject of food analysis. In the section on the sanitary examination of water, the author naturally gives some considerable space to the question of its bacteriological examination, and duly illustrates this part by a well-drawn plate and several diagrams. He does not, however, incline to taking a too high view of the value of the method as at present used, and the reasons he gives are so pointed that they seem worth reproducing *in extenso*, as follows:—

"1. The cultivation methods employed have rather developed the ordinary micro-organisms capable of cultivation at a low temperature.

"2. Pathogenic organisms are all best grown at, comparatively speaking, high temperatures—that is, from 20° to 40°.

"3. The liquefying bacilli and bacteria almost invariably destroy a gelatine cultivation, dissolving everything after a few days, whereas some pathogenic organisms require long cultivations to develop properly.

"4. Although it is possible to pick out particular colonies, and cultivate them thus isolated from others, such a process can only be adopted as a scientific investigation. Analysis of water must be reasonably speedy, but cultivations of this kind would take many weeks."

## LAW REPORTS.

AT Limerick Petty Sessions on Friday a grocer, trading in George Street, was prosecuted by the corporation, as the urban sanitary authority, for exposing for sale at his premises a quantity of margarine, the article not having been labelled as such, in accordance with the statute of last Session. Proceedings were instituted under the 6th section of the Act, and for the defence it was contended that the statute was faulty, inasmuch as the wording of the section under which the case was brought referred to "every person dealing in margarine in the manner described in the preceding section." In the preceding section there was not a single word in regard to persons dealing in margarine, the subject-matter relating only to *employés* and assistants. Mr. Connolly, law adviser to the corporation, said the contention for the defence had been made by the margarine promoters. In a book issued by them it was stated—and this was now relied on for the defence to this prosecution—that "the Protectionists and the Irish party were wide awake at 3 or 4 o'clock in the morning, and changed the name of the bill from butterine to margarine while the foolish virgins of Free Traders all slumbered well." The magistrates overruled the point raised for the defence, but subsequently, by a majority of five to four, dismissed the case on the ground that there was not sufficient evidence to establish the charge made against the defendant. The prosecution was the first under the Act in Limerick.

## APPOINTMENT.

MR. C. M. Aikman, M.A., B.Sc., F.I.C., F.C.S., Analytical and Consulting Chemist, 183, St. Vincent Street, Glasgow, has been appointed by the Municipal Authorities of Lanark Public Analyst for the Burgh.

ERRATUM.—In Messrs. Hehner and Richmond's paper on "The relation of specific gravity, fat and total solids, p. 27, sixth line from top, read "Fleischmann's plaster extraction formula" instead of "Morgan's formula."



# THE ANALYST.

APRIL, 1888.

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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A meeting was held on the 14th ult. at Burlington House. In the absence of Mr. Allen, the chair was taken by Dr. Vieth, Vice-President. The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened, it was announced that the following gentleman had been elected as a member:—

Mr. Gibson, analytical chemist, of Chicago.

The following papers were read and discussed:—

"Iodine Absorptions combining Weights, and Melting Points of certain Fatty

Analyses of Yeast," by Roland Williams.

"Analyses of Goose Fat," by W. C. Young.

"On Adulteration, and Pepper Analysis," by F. M. Rimmington.

Mr. Rimmington showed a number of samples of various kinds, in illustration of which they were microscopically examined by the members present, with much interest. His paper will be published in THE ANALYST for May, with a set of illustrations and specimens exhibited.

The next meeting of the Society will be held on Wednesday, the 11th April, at Burlington House.





These results prove that, in determining peroxide of hydrogen of 2-volume strength, in order to ensure reliable results, it suffices to employ 20 c.c. of a 10 per cent. solution of potassium iodide (or 2 grms. in 20 c.c. water), and 20 c.c. of 1 : 5 sulphuric acid. It will be seen that 10 c.c. of acid was insufficient.

Other experiments showed that, in cases where 10 c.c. of a 20 per cent. solution of potassium iodide was employed, less acid was required. Thus, using another solution of peroxide of hydrogen (10 c.c.)—

Amount of acid used.	N/10 hypo required.
10 c.c.	33.9 c.c.
20 c.c.	33.9 c.c.
30 c.c.	33.9 c.c.
40 c.c.	34.0 c.c.
50 c.c.	34.0 c.c.

By increasing the quantity of potassium iodide solution to 20 c.c., 30 c.c., 40 c.c., and 50 c.c. respectively the results were not altered, thus proving that 10 c.c. of 20 per cent. potassium iodide solution was sufficient quantity to use to ensure with 10 c.c. of 1 : 5 sulphuric acid the full decomposition of the peroxide contained in 10 c.c. of the solution under examination.

Many series of experiments were made with solutions of potassium iodide and sulphuric acid, each of varying strength, but it is needless to multiply results in view of my present object—viz., to indicate the quantities of potassium iodide and sulphuric acid which it is necessary to use in the practical estimation of peroxide of hydrogen.

I recommend, when the solution of peroxide of hydrogen is stronger, to dilute down with water to about 2-volume strength before conducting the estimation.

#### NOTES ON THE ESTIMATION OF MILK-SUGAR IN MILK BY MEANS OF THE POLARISCOPE.

By DR. P. VIETH, F.C.S., F.I.C.

(*Read at the Meeting, February, 1888.*)

TWELVE months ago I brought before you the results of a number of complete milk analyses, and made some observations with regard to the analytical methods employed. I then stated that the milk-sugar was determined by means of the polariscope, and that I followed the directions laid down by Schmoeger for preparing a clear whey. In the discussion it was remarked that the results might perhaps come out different and more correct if mercuric nitrate were employed for removing albuminoids and fat, instead of acetic acid and basic acetate of lead. The method of precipitating by means of mercuric nitrate has been described in THE ANALYST, XII., p. 196.\* I made some comparative experiments and found that the results were not materially influenced, but came out practically the same, whether the precipitation was effected by acetic acid and sub-acetate of lead, or by mercuric nitrate. However, I adopted the latter mode of procedure as the more simple and expeditious. According to Schmoeger, one has to precipitate with acetic acid, then add sub-acetate of lead, boil, cool down, replenish evaporated water, and filter again; while, when precipitating with mercuric nitrate, the latter is mixed with the milk and the mixture brought on a filter, when clear whey runs off ready for polarisation.

\* See also "Foods and Food Adulterants, U.S. Department of Agriculture, Washington, p. 133."

The whey thus obtained is always perfectly bright, not only when poor or normal milks are under examination, but also when rich milks and even cream of the greatest possible richness are operated upon. I have experimented with cream containing upwards of 50 per cent of fat, and still got quite clear whey. This is rather surprising, considering that such a cream contains about 2.25 per cent. of proteids only, an amount which would appear altogether inadequate to encase on precipitation 50 per cent. of fat, that is more than twenty times its own weight and about thirty times its own volume. I thought it not improbable that the mercuric nitrate might perhaps have a direct action on the fat globules, such as rendering the liquid fat, of which they are composed, solid, and that in consequence of an alteration of this kind they might adhere to the coagulum and not run through the pores of the filter. But microscopic examination did not reveal any change in the shape and appearance of the fat globules.

There is one difficulty when operating on cream rich in fat, viz., to get a sufficient amount of liquid for polarisation. The mixture of cream and mercuric nitrate solution does not retain the character of a liquid, but attains that of a pulpy mass, its consistency increasing with the increasing percentage of fat. I am in the habit of taking 50 c.c. for the milk-sugar determination. Experimenting with a thin cream containing about 25 per cent. of fat, not sufficient whey drained through the filter to fill a 200 millimeter tube, and I had to use slight pressure to get the required quantity. With richer creams the difficulty is getting greater, and cream containing upwards of 50 per cent. of fat gave a few drops of whey only when the filter in the funnel was subjected to a pressure of two pounds. In such cases then it is necessary to dilute the cream with, say, an equal volume of water before precipitation.

Schmoeger has found that the volume of the precipitate caused by the addition of acetic acid to 100 c.c. of milk is between 5 and 6 c.c., and in the article on the employment of mercuric nitrate for preparing the whey for polarisation it is stated that the precipitate from 60 c.c. of milk occupies the space of 2.4 c.c., equal to 4 c.c. from 100 c.c. of milk. Such statements and directions for working the process in question based upon them are rather misleading. Schmoeger worked upon milk containing something like 3 per cent. of fat, while in the other case the amount of fat varied from 3 to 5 per cent. Supposing a milk contains 4 per cent. of fat, this alone would occupy the space of 4.3 c.c. and to it would have to be added the volume of the precipitated proteids. It needs not many words to demonstrate the absurdity of once for all fixing a definite correction for the volume of the precipitate, as if this were a constant factor. The volume of the precipitate must of necessity vary with the varying quality, and more especially richness of milk.

It is a too well-known fact, to be dwelt upon in this place, that of all the milk constituents the fat is the one which varies between the widest limits. The percentage amount of the non-fatty solids is not subjected to such wide variations; and as the non-fatty solids consist of three compounds or groups of compounds, as, moreover, it has been proved, that these three constituents are in normal milks always present in very near the same relative proportion, we may expect the proteids to vary very little indeed in their percentage amount. According to my experience the limits for proteids are about 3.5 and 4.0 per cent. I believe I cannot be very wrong in assuming that this



amount of proteids, if precipitated in 100 c.c. of milk, will occupy the space of about 3 c.c. If then 3 c.c. of mercuric nitrate solution were added to 100 c.c. of milk, free of fat, or 1.5 c.c. to 50 c.c., the filtrate would for polarisation be equal to the milk employed. The reading of the polariscope would represent percentage of sugar by volume, which can easily be transformed into percentage by weight, by referring to the specific gravity. If, however, as always will be the case, fat is present, another calculation becomes necessary. The percentage amount of fat is to be enlarged in the proportion of 93 to 100, the product being percentage of fat by volume, and the amount of sugar read off to be reduced in the proportion of hundred times specific gravity of milk (specific gravity of water = 1) to 100 minus volume of fat. The figure thus obtained represents the amount of crystallised milk-sugar, and is to be reduced by one-twentieth of its value for water of crystallisation.

An example will perhaps show more clearly the mode of proceeding. Supposing we have to deal with a milk of specific gravity 1.0325, and containing 3.72 per cent. of fat. To 50 c.c. of this milk is added 1.5 c.c. of mercuric nitrate solution; the whey on polarisation indicates 5.1 per cent. milk-sugar. The necessary calculations are then as follows:—

$$\begin{array}{rcl} 93 : 100 = 3.72 : x. & & x = 4.0. \\ 103.25 : 96 = 5.1 : x. & & x = 4.74. \\ & & 4.74 - .24 = 4.50. \end{array}$$

In case an excessive amount of fat should be present, the percentage quantity of proteids will naturally be depressed to some extent, and the quantity of mercuric nitrate solution may be reduced accordingly. But, even if this is not done, the error will be very slight indeed, as may be gathered from the fact that cream with 50 per cent. of fat still contains about 2.25 per cent. of proteids.

#### AN INSTRUMENT FOR CALCULATING MILK RESULTS.

By H. DROOP RICHMOND.

(*Read at Meeting, February, 1888.*)

THIS instrument, which is based on the relation between fat, solids not fat, and specific gravity in milk, indicated in a recent paper by Mr. Hehner and myself (*ANALYST*, xiii., p. 26), consists of a slide rule, on one side of which a scale (1 division = 1 inch) represents total solids, on the other a scale (1 division = 1.164 inches) shows fat, while on the slide specific gravity is indicated (1 division = .254 inch); these numbers show the relation according to the formula  $T - .254 G = 1.164 F$ . The instrument is very simple to use; the lines indicating the total solids and gravity found by analysis being placed together, the fat is immediately read off by an arrow on the other side, or, the arrow being placed against the line indicating fat found, the total solids is coincident with the gravity found, thus saving a large amount of time besides totally eliminating all chance of error of calculation; to analysts having many milks it will be especially useful.

In the construction (which has been undertaken by Messrs. Watson and Sons, of 313, High Holborn) it has not been found practicable to include the small correction recommended for poor skim milks, but the error introduced by this will only in extreme cases amount to .08 per cent, and is usually negligible.

## ON A SAMPLE OF "NAVY GREEN" PAINT.

BY BERTRAM BLOUNT.

*(Read at the Meeting February, 1888.)*

A SAMPLE of paint called "Navy Green" recently came into my hands, and as it seemed to be of somewhat unusual composition, I thought that a few words concerning it might not be without interest to our Society.

Examination of the paint soon showed that it did not consist, like the common kinds of green paint, of some cheap white pigment, such as barium sulphate, calcium sulphate, or chalk, coloured by a mixture of chromate of lead and Prussian blue.

Barium sulphate and chromate of lead were certainly present, but no Prussian blue, so that it remained to ascertain what was the blue constituent.

A qualitative analysis of the paint after ignition, to get rid of the oil with which it had been ground, proved the absence both of copper and cobalt, and in another portion of the paint, which had been extracted with petroleum ether, a search made for chromic oxide, ultramarine, and indigo, met with a negative result in each case.

Having thus fairly well exhausted the list of likely substances, I renewed the examination, as if it were a body about which nothing was known.

The dry extracted paint was treated with water, which immediately became a beautiful blue colour with a greenish tinge, and the insoluble matter was left a pure yellow, shewing that a separation between the chromate of lead and the blue colouring matter had been effected.

The solution was filtered and the filtrate concentrated; colourless crystals were observed to separate as the concentration proceeded, and on reaching dryness were present in considerable quantity, standing out quite distinct from the rest of the residue, which was blue and amorphous. It was found that the blue substance was soluble in alcohol while the colourless crystalline body was not; they were accordingly separated by means of it.

The colourless crystalline substance proved to be merely calcium sulphate.

The proportion of blue colouring matter was then determined by a method practically identical with that used for its qualitative isolation, and found to be .24 per cent. on the oil-free paint, the actual numbers being as follows:—

4.2687 grms. of paint yielded .0102 grms. of the colouring matter; this gave on ignition .0032 grms. of ash. I do not suppose that the whole of this ash was from the colouring matter itself, but that rather it consisted of traces of metallic salts, which had escaped elimination in the extraction of the blue substance, the more so because it contained calcium sulphate, which body was abundantly present in the first extract with water, as was mentioned above.

I then extracted the blue substance from the greater part of the small quantity of paint at my disposal, and tried to identify it with some known colouring matter.

*It was found to be soluble in:—*

Water.—Solution neutral to litmus and methyl orange; faintly acid to phenolphthalein.

Alcohol.—Solution lighter in tint than the aqueous one for the same strength.  
Glycerin.



Glacial acetic acid.—Left apparently unaltered on evaporation.

It was found to be insoluble in :—

Ether.	Chloroform.	Turpentine.
Carbon disulphide.	Benzene.	Petroleum Ether.

Heated with soda-lime.—Slightly alkaline fumes, but the smell was not ammoniacal.

Treated in alcoholic solution with chloroform and caustic potash.—No smell of an isocyanide.

Treated in aqueous solution with iodine dissolved in potassium iodide.—Nothing was thrown down, unless the solutions were very concentrated, when a scanty dark-brown precipitate formed on standing. It was then examined by the tables drawn up by E. Weingaertner, which are a development of the method proposed by Otto N. Witt for the separation of artificial colouring matters.\*

† Pursuing the plan laid down in these, the substance came out under the head of the azo colours, which together with erythrosin and tartrazin form one group, but in the list of them given along with the tables none occurs which is blue, except erythrosin which is said to be a reddish—instead of a greenish—blue, and the reactions of which clearly distinguish it from this substance; for example, erythrosin gives off iodine when heated with strong sulphuric acid, whereas no halogen is evolved from this substance even when manganese dioxide is added. No definite result being obtained by the use of these tables, the behaviour of the substance with various reagents was observed.

The aqueous solution was treated with :—

Sodium chloride.—None of the colour was precipitated even on saturating its solution with the reagent.

\* Jour. Soc. Dyers and Colourists, April, 1887.

† The following contains as much of these tables as is necessary for the substance under consideration :—

To the aqueous sol. of the colouring matter a few drops of a reagent, consisting of tannin 1 part, acetic acid 1 part, water 10 parts, are added, and the mixture heated. If no precipitate appears (as was the case with this substance), examine by Table II., which is given as far as it relates to this substance, below.

TABLE II.

The aqueous solution is treated with zinc powder and hydrochloric acid, or zinc powder and ammonia.

The solution is decolorised.

Throw on to a piece of filter paper.

The original colour  
reappears.

The original colour  
does not reappear.

Heat the colouring matter on platinum foil.

Deflagration without  
coloured vapours.

The solution becomes brown-red.

Quiet combustion with  
coloured vapours.

Heat a piece of unmordanted  
calico in the aqueous solution.

The dye is fast  
to warm soaping.

The dye is not fast to  
warm soaping.

= { Azo colours.  
Tartrazin.  
Erythrosin.

The only respect in which the substance deviated from the course indicated in the above table is not giving off coloured vapours when heated.

Hydrochloric acid.—Blue colour destroyed leaving only a yellowish tint ; on adding ammonia colour restored, but bleached again by excess. On acidifying the ammoniacal solution with acetic acid the colour was again restored, and was unaffected by excess of the reagent. The colour also reappeared on evaporation to dryness.

Caustic potash.—Blue colour destroyed ; in a concentrated solution a slight flocculent colourless precipitate forms on standing. The blue colour is restored by acidulating with acetic acid even though the potash be used boiling and in excess.

Ammonia.—Blue colour destroyed ; partly restored on heating or on neutralising with hydrochloric acid ; completely restored by the addition of acetic acid in excess.

Sulphuretted Hydrogen.—Colour unaffected even on boiling.

Ammonium sulphide (colourless).—Colour bleached.

Sodium sulphite.—Colour at once bleached. On adding acetic acid and warming the colour returned, but on cooling faded again. This appearance and disappearance of the colour could be repeated several times. On boiling the solution to drive off the  $\text{SO}_2$  the colour became permanent on cooling.

Sodium thiosulphate.—Slowly and imperfectly bleached even on long boiling.

Stannous chloride.—Somewhat bleached ; on standing wholly decolourised.

Bromine water.—Bleached ; colour not restored by stannous chloride.

Calcium hypochlorite.—Immediately bleached.

Nitric acid (dilute).—Became green, and on standing yellowish ; evaporation in vacuo gave no crystalline product. If neutralised with ammonia and then acetic acid added in excess the blue colour was restored.

Nitric acid (conc.)—Behaved as with dilute acid, but on heating became colourless, and the blue could not be restored by treatment with ammonia and acetic acid.

Other weak acids, such as oxalic, citric, and boric, behaved like acetic and did not affect the colour.

Albumen (white of egg).—The colour was precipitated together with excess of albumen on heating to the temperature necessary for coagulation. If acetic acid was added to perfect the precipitation of the albumen the colour was not thrown down.

Lead acetate.—Apparently no reaction ; no product could be detected even on evaporation in vacuo and examination under the microscope.

Basic lead acetate.—Colour completely thrown down as an amorphous greenish-blue precipitate. This precipitate was easily soluble in acetic acid, and it was found that the lead could be removed by sulphuretted hydrogen, and the colouring matter recovered.

Silver nitrate.—Slight reduction ; when evaporated in vacuo no crystalline compound could be detected under the microscope.

Mercuric chloride.—No apparent change in the cold or on boiling ; on evaporation a bluish-green, amorphous precipitate was formed.



Platinic chloride.—Beautiful yellow crystals, shaped like a four-pointed star, were formed on evaporation in vacuo. They were well seen under a  $\frac{4}{10}$  in. objective.

Sulphuric acid conc. (on the dry substance).—Dissolved with the formation of a yellow colour, becoming greenish on dilution.

*Behaviour of the substance as a dyestuff* :—

- (1) Without a mordant.
  - On cotton .. Not fast to cold water.
  - On silk } .. Not fast to boiling water.
  - On wool }
- (2) With alumina as a mordant.
  - On cotton .. Not fast to cold water.
  - On silk } .. Not fast to boiling water.
  - On wool }
- (3) With albumen as a mordant.
  - On cotton .. Not fast to cold water.
  - On silk } .. Not fast to boiling water.
  - On wool }
- (4) In a bath made slightly acid with acetic acid.
  - On cotton .. Not fast to cold water.
  - On silk .. Fast to boiling water; fairly fast to warm soaping.
  - On wool .. Fast to boiling water; not fast to warm soaping.

I have been unable, from lack of material, to prepare and purify in quantity sufficient for an ultimate analysis, the only crystalline compound I have obtained, viz., the platinum salt, nor can I throw light on the proximate composition of the body, but hope that the empirical reactions I have detailed may suffice for its identification.

I may say that having failed to find a description of a colouring matter which allied with it, I sought to learn its commercial name from the vendor, but found that he only knew it as "Mid Zinc Green," under which lucid title he received it from a German manufacturer.

(*Conclusion of Societies' Proceedings.*)

#### ON ADULTERATED LARD.

By STEPHEN P. SHARPLES, STATE ASSAYER, MASSACHUSETTS.

In the interest of pure food the testimony of Mr. N. K. Fairbanks and Mr. Webster, before the Committee on Agriculture of the United States Senate, should have wide circulation.

They testified "that all of the lard on the market marked 'Prime Family Refined Lard,' 'Choice Refined Lard,' and other brands of this nature is mixed with more or less beef stearin and cotton-seed oil."

As it is well known that cotton-seed oil is a semi-drying oil, having strong siccative properties at the temperature of 212° F., this admixture unfits the lard for many uses.

It is impossible to make good biscuits with such a compound, as they rapidly become rancid.

The above gentlemen represent two of the largest firms in the so-called "refining" business in Chicago.

The refining of lard consists solely in adulterating it with cotton-seed oil and oleo-stearin.

These mixtures may be easily detected. The usual tests for detecting cotton-seed oil in olive oil answer every purpose. Bechi's test, as given in THE ANALYST, gives good results. Lard is without action on the solutions used. Nitric acid of 1.35 specific gravity gives only a faint colour with pure lard, with lard adulterated with cotton-seed oil it gives a colour more or less intense, varying with the quality and quantity of the oil used. For the beef stearin the best test is that proposed by Dr. Belfield, of Chicago, as follows:—The suspected lard is dissolved in ethylic ether, so as to form a nearly saturated solution. This is best done in an ordinary five-inch test tube, which should be about two-thirds filled with the mixture. The top of the tube is then loosely stopped with cotton wool, and it is placed in a quiet place, at a temperature of about sixty degrees, and allowed to stand until crystals commence to form. These crystals are removed from the tube with a dipping tube and placed on a microscope slide; they are quickly covered with a thin cover-glass, pressed enough to flatten the grains, and then examined with a quarter-inch objective.

Pure lard gives large flat plates with well-defined oblique terminations. These are sometimes in radiated groups, but often occur singly. Beef fat always crystallises in radiating tufts, often resembling wheat-sheaves, and the crystals are either pointed or else have nearly square terminations. They are always, however, much more slender than the lard crystals.

Watering lard has almost become one of the lost arts, only one sample from nearly a hundred examined had any marked amount of water; this one, however, had over forty per cent. It was kept in combination by means of an alkali.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

AN EASY METHOD OF FINDING THE SPECIFIC GRAVITY OF LIQUIDS. A. B. TAYLOR, *Am. Journ. Pharm.*—A new application of an old rule has suggested a method of finding the specific gravity of liquids, which the author has never seen mentioned, and which from its simplicity and great ease of application seems worthy of publication. It is known that the weight of a body is to its specific gravity as its loss of weight when immersed in a liquid is to the specific gravity of that liquid. For example:—200 grains of citric acid (sp. gr. 1.60) lose in weight 115 grains when weighed in oil; and as 200 is to 1.60, so is 115 to .920 the sp. gr. of the oil. Now if we make the weight of the citric acid the same number of grains as its specific gravity our formula becomes—as 1.60 is to 1.60, so is the loss in weight of the citric acid when weighed in oil to the specific gravity of the oil; or, in other words, the loss of weight is equal to the specific gravity; from which we deduce the following general rule:—

The specific gravity of a liquid is equal to the loss of weight (in grains) sustained by a solid body when immersed in the liquid, the weight of the solid being equal (in grains) to the specific gravity. Hence it is necessary only to weigh the solid in the liquid, and its loss gives at once the specific gravity of the liquid. Taking the preceding



example:—If 200 grains of citric acid lose 115 grains, 1·6 grain will lose ·920 grain, and this loss is equal to the specific gravity of the oil. In practice the weight of the solid might be 10 or 100 times the weight of its specific gravity, care being taken to put the decimal point in the right place in the final result. As perhaps one of the most desirable solid bodies to use, the author suggests a piece of aluminium weighing 256 grains; the specific gravity of that metal being 2·56. If upon trial its specific gravity should vary from these figures, its weight should be made to correspond. For liquids having greater specific gravity than 2·56 it would be necessary to use a heavier solid than aluminium.

W. H. D.

DETECTION AND ESTIMATION OF ROSIN OIL IN MINERAL OIL. L. STORCH. *Ber. Chem. Ind.*, ix., 93.—To test for rosin oil the author takes 2 c.c. of the sample and shakes it up with 1 c.c. of anhydrous acetic acid, and gently warms it. He then cools, removes the acetic liquid by means of a pipette, and adds to it a drop of strong sulphuric acid, when a brilliant red colour is immediately produced if rosin oil be present. Owing to the presence of cholesterin in many fatty oils they give a similar reaction, and, therefore, the absence of such oils must be first thoroughly proved. For the estimation of rosin oil in mineral oil (in the assured absence of fatty oils) the author proceeds as follows:—10 grammes of the sample are taken, gently warmed with 50 c.c. of alcohol of 96 per cent., the whole is well shaken and allowed to cool. The liquid having separated into two layers the alcoholic stratum is transferred to a tared flask, and the surface of the petroleum layer having been washed with a few c.c. of alcohol, and the washings added to the bulk, the flask is placed on the water-bath. The contents are thus gently boiled down and the evaporation is continued until the residue in the flask is observed to be free from bubbles, when it is cooled and weighed. This residue is rosin oil with a certain amount of mineral oil still remaining (residue A). This residue is then extracted with ten times its weight of similar alcohol exactly in the same manner, and another weighed residue (B) is obtained. This second residue is now almost free from mineral oil. By now deducting the weight of alcohol used in experiment B from that taken for A, and also deducting residue B from residue A, it is evident that the exact solubility of the mineral oil in the alcohol employed can be determined, and a final correction made. For example, in a case where 10 per cent of rosin oil was actually present, 50 grammes of alcohol were used for A and 15·5 grammes for B; giving a difference of 34·5 grammes. Residue A weighed 1·5136, and residue B weighed 1·1584; difference = ·3552 grammes of mineral oil, dissolved by 34·5 grammes of alcohol. Therefore, the 15·5 grammes of alcohol used for B really ought to have dissolved ·1595 of mineral oil. This correction, deducted from residue B, leaves ·9989 gramme for the weight of rosin oil present, or over 9 per cent of the weight of the sample taken for analysis. Had residue B been taken without correction it would have shown 10·32 per cent. so that the truth lies between the two results.

W. H. D.

ESTIMATION OF TANNIN. F. GANTTER. *Zeitschr. f. Anal. Chemie*, vol. vi., 1887.—The usual process is to titrate a known volume of the solution with permanganate, and then another portion, after first removing the tannin by means of hide powder. The

use of this substance being inconvenient a proposal has been made to replace it by ferric acetate. The author has made a series of experiments to test the accuracy of the new process. If the process with the hide powder is at all trustworthy (which is, however, questionable) the new proposal cannot be entertained. When the materials are very poor in tannin, and when, in consequence, a large quantity is weighed out, the differences are not so very great—about 1 per cent., but in the case of rich materials the results differ from 5 to 11 per cent.

L. DE K.

ESTIMATION OF LEAD IN ALLOYS OF TIN. Y. SCHWARZ. *Chem. Zeit.* 4, 1888.—

One gram. of the thinly flattened alloy is gently heated with 20 c.c. of concentrated hydrochloric acid. The tin is generally dissolved in about half an hour. Without troubling about any undissolved spongy metallic mass, bromine is added until the liquid turns yellow, when everything will have dissolved. The excess of bromine is now boiled off and the liquid diluted up to 100 c.c. This, after cooling, is slowly poured into a solution of 40 grammes of crystallised sodic sulphide in 150 c.c. of water. After the plumbic sulphide has settled, it is filtered off and washed with weak solution of ammoniac sulphide (1—10). The filter and contents are now heated, with the usual precautions, in a porcelain dish with a mixture of nitric and sulphuric acid, to make the plumbic sulphide into sulphate, which may then be washed with proof-spirit and collected and weighed as usual. It should be completely soluble in an alkaline solution of ammoniac tartrate. If it leaves a residue of stannic oxide, this must be weighed and deducted. Its weight should not exceed a few milligrammes.

L. DE K.

DETECTION OF ADULTERATED GROUND BONES. E. HEIDEN. *Chem. Zeit.*, ii., 1328.

—Ground bones are frequently made from bones that have been deprived of their gelatine by boiling. In this way the nitrogen is lowered, and the phosphoric acid unduly increased. To remedy this the manufacturer often adds scraps of hoofs and similar nitrogenous matters and, at the same time he puts in sand or gypsum to bring the amount of the phosphoric acid within normal limits. The author has applied a similar process to that employed for the detection of alum in flour, viz., by shaking the suspected sample up with chloroform. When this is done the genuine bone powder sinks to the bottom, while the spurious additions float. No genuine sample of ground bones will show more than 5 per cent. of floating particles.

W. H. D.

ANALYSIS OF BLACKING. V. HÖBLING. *Zeitschr. f. angew. Chemie.* No. 1.—

Analyses of apparently very simple mixtures, like blacking, give, as a rule, more trouble to analysts than delicate investigations. It will, therefore, no doubt, be interesting to them to read the author's process. A weighed portion of the sample was boiled with water; the residue collected on a weighed filter and washed until the filtrate was colourless. After drying and weighing the filter was exhausted in a Soxhlet's apparatus with petroleum spirit. After evaporating the latter the residual fat was weighed. The filter and contents were burnt to ash, and the total carbon was thus found by difference. This carbon is a mixture of carbon derived from animal charcoal, and ditto formed by the action of the sulphuric acid on the sugar. A fair idea of the quantity of the first



may be got by the estimation of the calcic phosphate as the ratio between tricalcic phosphate and carbon in bone-black is generally as 9 to 1.

The ash was estimated in a separate portion of the sample and further analysed by the usual processes, the chief constituents being lime, soda, iron, phosphoric and sulphuric acids. The fat was saponified with potash and the mineral oil extracted by agitating the fluid with petroleum spirit. By titrating back the excess of alkali the saponification equivalent of the fat was obtained, which threw some light upon its nature. The watery extract was diluted up to a definite bulk, and in an aliquot part, the sugar was gravimetrically estimated by Fehling's solution. Glycerin was first qualitatively tested for as follows: 2 drops of the liquid were mixed with 2 drops of phenol and 2 drops of sulphuric acid, then heated up to  $120^{\circ}\text{C}$ ., and, after cooling, dissolved in ammonia. If glycerin is present the solution will be of a carmine red colour. The quantitative estimation is best performed by the process of Becke and Mayer (*Zeitschr. f. anal. Chemie*, 1880, 291). If more than 1 or 2 per cent. of iron has been found this points to the use of ink (tannate of iron). A portion of the watery fluid is acidified with hydrochloric acid, and repeatedly agitated in a separating funnel with acetic ether, which dissolves the tannin. This solution is then poured on some water, and the ether expelled by heat. If tannic acid should be present it is best estimated by the well-known process with potassic permanganate. The result of the analysis of one of the samples submitted to the author was as follows:—

Water, 12.36; free sulphuric acid, 1.38; glycerin, 3.45; invert sugar, 10.49; mineral oil, 4.02; neatsfoot oil, 4.89; total carbon, 12.63; lime, 16.06; sulphuric anhydride, 10.04; phosphoric anhydride, 12.65; oxide of iron, .84; soda, 5.78 parts.

The phosphoric anhydride corresponds with 32.82 bone-black, or 3.28 carbon. This leaves 9.35 carbon, corresponding with 28.05 of sugar, thus making a total of 38.54 of sugar. Taking the composition of molasses as constant (24.7 per cent. of sugar), this will then represent 156.03 parts of original molasses. To char the sugar to the extent found 44.22 parts of sulphuric acid must have been required (according to the equation:  $\text{C}_6\text{H}_{12}\text{O}_6 + 2\text{H}_2\text{SO}_4 = 5\text{C} + \text{CO}_2 + 2\text{SO}_2 + 8\text{H}_2\text{O}$ ), the total amount of the acid, therefore, being 44.22 parts.

The soda (which in practice is added after the other ingredients have been mixed, to stop further action of the acid), is equivalent to 9.88 parts of caustic soda. The composition of the sample may, therefore, be fairly given as: bone-black, 33; molasses, 156; oil of vitriol, 44; mineral oil, 4; neatsfoot oil, 5; glycerin, 3.5; caustic soda, 10.

The fat is sometimes decomposed into fatty acid and glycerin. Corresponding quantities of these must be calculated to fat. The author does not, of course, claim absolutely accurate results.

L. DE K.

ESTIMATION OF STARCH BY MEANS OF IODINE. F. SEYFERT. *Zeitschr. f. angew. Chemie*. No. 1.—The author found the true formula of iodide of starch to be  $(\text{C}_{24}\text{H}_{40}\text{O}_{20})_n\text{I}_7$ , and has based upon this fact a volumetric estimation. 1 gramme of the finely-ground flour is heated with about 150 c.c. of water for two hours in a water-bath, which will cause the starch to gelatinise. After cooling the liquid is introduced into a 500 c.c. flask and mixed with 50 c.c. of deci-normal iodine. 20 c.c. strong hydrochloric acid are

added, and the whole diluted with water up to the mark. After the iodide of starch has completely settled an aliquot part of the clear liquid is taken off with a pipette and titrated with sodic hyposulphite. The difference in iodine is multiplied by 4.37 to get the corresponding amount of starch. The test experiments are encouraging. In one sample of dried potato-flour the amount of starch found was 73.9 per cent. The same sample, when analysed by the baryta process, gave 73.6 per cent. L. DE K.

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ESTIMATION OF MANGANESE IN CAST IRON. C. REINHARD. *Zeitschr. f. angew. Chemie*, No. 4.—The author recommends the following process for samples, poor in manganese, but rich in silicon. About 3 grammes of the sample are dissolved in about 40 c.c. HCl, with addition of some potassic chlorate. After evaporating to about 20 c.c. the liquid is diluted with water, filtered into a 500 c.c. flask, and the residue well washed. 15 c.c. of nitric acid are added, and the liquid boiled for some time. Zinc oxide suspended in water is now added until the iron is completely precipitated, and after cooling, the whole is made up to the mark, and filtered. An aliquot part (say 250 c.c.) is now mixed with a little sodic acetate, and boiled with a little zinc oxide and bromine water. The manganese separates as  $MnO_2$ , and is finally titrated with oxalic acid as usual. L. DE K.

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ESTIMATION OF CAUSTIC ALKALIES IN PRESENCE OF ALKALINE CARBONATES. ISBERT AND VENATOR. *Zeitschr. f. angew. Chemie*, No. 4.—The usual plan is to first titrate with standard acid, and then again, after precipitating the carbonate with baric chloride. As, however, the percentage of caustic alkali is generally found too low by this process the authors adopt the following plan: A weighed or measured quantity of the sample is diluted and titrated in the cold with normal acid until the fluid turns distinctly yellow, an alcoholic solution of rosolic acid being used as indicator. The amount of the caustic alkali is now calculated. To find the quantity of alkaline carbonate, the same fluid must now be boiled, with occasional addition of normal acid, until the yellow colour no longer changes to red. From the extra amount of acid used, the alkaline carbonate is calculated as usual. L. DE K.

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A NEW PROCESS FOR THE ESTIMATION OF ALCOHOL. B. RÖSE. *Zeitschr. f. angew. Chemie*, No. 2.—If potassic permanganate is added to alcohol, mixed with dilute sulphuric acid, an imperfect oxidation takes place, even if the mixture is heated. If, however, very dilute alcohol is first mixed with large excess of permanganate, and then suddenly with about one-third of its volume of sulphuric acid, the alcohol instantly and completely oxidises to carbonic anhydride and water. Water may now be added, and the excess of permanganate titrated back with potassic tetra-oxalate. From the amount of permanganate decomposed, the alcohol can be readily calculated, 8.244 grammes of permanganate equal 1 gram. of alcohol. The test analyses, four in number, are very satisfactory. Further experiments are promised. L. DE K.



## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

A RIGHTHANDED POLARISING SAMPLE OF HONEY. R. BENSEMANN. *Zeitschr. f. angew. Chemie*, No. 4.—The sample, which dissolved in water suspiciously clear, had the following composition:—water, 22.61, ash, .09, levulose and dextrose 64.33, sucrose, 12.59. Rotation = + 3.74. The author, therefore, felt naturally inclined to condemn the sample, as being in fact nothing else but badly-made invert sugar, or a mixture of real honey and cane-sugar; but after strong protests of both the dealer and the beekeeper, he investigated the matter on the spot. Samples taken directly from the comb practically showed the same composition. Unless cleverly imposed upon, the author can only account for this by the fact that the bee farm is situated very close to a sugar refinery, where the bees have plenty of opportunity of feeding on the sugar, and may lack sufficient acidity (formic acid?) to completely invert the sugar. L. DE K.

APPLICATION OF THE DIPHENYLAMINE TEST TO WINE. E. POLLAK. *Chem. Zeit.*, i., 1465.—The author has applied this test to a series of Rhenish wines, and he finds that no natural wine of this class contains nitrates in a sufficient amount to show any marked reaction with this test. The inference, therefore, is that if a blue colour be obtained in a wine when tested with diphenylamine, the article under examination is not natural but has been watered or otherwise mixed with nitrates. W. H. D.

ESTIMATION OF TOTAL SOLIDS AND FAT IN MILK AND BUTTER BY MEANS OF WOOD-PULP. F. GANTTER. *Zeitschr. f. Anal. Chemie.*, vol. vi., 1887.—The author recommends the following process for the analysis of milk:—Wood-pulp, such as is used in paper-mills, is dried and freed from fatty and resinous impurities by extraction with petroleum-ether. About 2 grammes of it are put into a small dish, provided with a small glass rod and also with a good fitting lid, and the whole is then dried to constant weight at 105°C. A small beaker, holding about 6 grammes of milk, is now weighed, and after pouring the contents on to the wood-pulp, re-weighed. The dish is now placed on the open waterbath and the contents well manipulated with the rod. When the mass is apparently dry the dish is put again in the air-bath and dried to constant weight, which is usually the case in one hour and a half, the whole operation taking from two to three hours.

It will now be found possible to remove the dried mass from the dish without the slightest loss, to a Soxhlet's apparatus. Two hours' extraction with petroleum-ether will be found sufficient. The results obtained by repeated analyses of milk, cream, and skim milk are wonderfully concordant. Butter and other fats may also be rapidly analysed in this manner. For 5 grammes of the fat about 3 grammes of wood-dust will suffice. L. DE K.

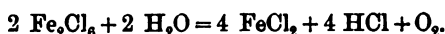
## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

ASSAY OF COLCHICUM SEED.—*Jour. de Phar. et de Chem.* A. KREMEL.—To determine the amount of colchicine, 20 grams. of the unbruised seeds are exhausted with

alcohol, in a displacement apparatus. After boiling for two hours, the alcoholic liquor is poured into an evaporating dish, with the alcohol used in washing the receiver, and 25 ccm. of water. The residuum after evaporation—10 to 15 ccm.—is filtered and exhausted with chloroform, which takes up the colchicine. It is again dissolved in chloroform, which is finally evaporated in a water-bath. The chloroformic extract is treated with water to disassociate the combination  $C_{22}H_{25}NO_6 + 2CHCl_3$ , which has formed; and is then evaporated to dryness. W. H. D.

ESTIMATION OF CARBONIC ACID IN MINERAL WATERS. W. BORCHERS. *Zeitschr. f. Anal. Chemie*, vol. vi., 1887.—The author published in 1878 his process for the estimation of combined, semi-combined, and free carbonic acid in waters.\* The two latter were estimated by simply boiling the sample and absorbing the liberated  $CO_2$  in suitable tubes. The combined  $CO_2$  was then expelled by hydrochloric acid, and also estimated. The process did not, however, work well if the water contained much sodic or potassic bicarbonates, as the excess of carbonic acid is but slowly expelled from these compounds. This may, however, be overcome by first adding to the water a little baric chloride. Baric bicarbonate is formed, which quickly loses its excess of  $CO_2$  on boiling. L. DE K.

DELICATE TEST FOR MORPHINE. J. L. ARMITAGE. *Pharm. J.* 924, 127.—When a solution of ferric chloride is added to a solution of a salt of morphine a bluish-green coloration is produced. At the same time, in consequence of the reducing power of morphine, a part of the ferric chloride is reduced to the ferrous condition, as shown in the following equation:—



There is, however, a point of dilution (apparently about 1 in 2,000), at which the coloration is imperceptible, though the reaction represented in the above equation appears to take place even in the most dilute solutions. On this reaction is based a delicate test for morphine and its salts in solution, for if such a solution be treated with ferric chloride and then with ferricyanide of potassium, the latter will interact with the reduced ferrous chloride with the formation of Turnbull's blue, which appears either as a deep blue precipitate or greenish-blue coloration, according to the strength of the solution of the alkaloid. In a solution of one part of a salt of morphine in 20,000 parts of water, the coloration is intensely green, and in a solution of one part in 50,000, the shade is still deep, though lighter. A solution of 1 in 100,000 gives the same result on standing a few moments, the coloration, even in this dilute solution, being unmistakable. Other reducing substances would, of course, give the same results, but in the absence of such substances the above is a delicate confirmatory test for the presence of morphine. Several other alkaloids submitted to the above test did not give the coloration. This reaction might be made the basis of a colorimetric process for the approximate estimation of morphine in very dilute solutions. W. H. D.

\* *Jour. f. Pract. Chemie*, 17.



## REVIEW.

ORGANIC ANALYSIS: A MANUAL OF THE DESCRIPTIVE AND ANALYTICAL CHEMISTRY OF CERTAIN CARBON COMPOUNDS IN COMMON USE, FOR THE QUALITATIVE AND QUANTITATIVE ANALYSIS OF ORGANIC MATERIALS, COMMERCIAL AND PHARMACEUTICAL ASSAYS, THE ESTIMATION OF IMPURITIES UNDER AUTHORISED STANDARDS, FORENSIC EXAMINATIONS FOR POISONS, AND ELEMENTARY ORGANIC ANALYSIS. By ALBERT B. PRESCOTT, Ph.D., M.D., Director of the Chemical Laboratory in the University of Michigan, etc. New York: Van Nostrand.

THIS is the American "Allen," and, on seeing the title, some of our readers will doubtless expect a critical comparison of the two works. In this they will, however, be disappointed, because, after a most careful study of both books (with, we confess, such an attention) we have seen that each has its own idiosyncrasies and shortcomings, while, at the same time, each has its own specialties and perfections. For example, the very first thing that strikes the reader on opening this book is that the author has adopted the dictionary arrangement, as distinguished from the classified one followed by our English author. On this point alone, when two such doctors differ, who shall decide? For Dr. Prescott's method of putting the matter it may be said that one can at once find what he wants to read, while Mr. Allen may reply that by the alphabetical method, all connection between the various members of the same groups of bodies is apt to be severed. Although the actual contents of both works cover pretty nearly the same ground, yet the two authors go to work so differently that the whole of Dr. Prescott's book is compressed into one volume of 520 pages. Here again opinions will differ, some preferring the terse diction and frequent absence of detail, characterising the American, while others will like the more extended and precise style of the British author. Which book a man would buy will, therefore, evidently depend on his own taste; but we ourselves are sufficiently patriotic to give the preference to our home production, as a more thorough and compendious treatment of the subject, so far as it has yet developed itself in the two volumes at present published. At the moment, Dr. Prescott's book comes before us complete, while we still await the conclusion of Mr. Allen's work, and, besides this, Dr. Prescott has the advantage in having been better able to present us with the recent advances on all the subject than is in the power of our English author, whose volumes only appear at such long intervals that, in the present days of such rapid extension of the powers of analysts in dealing with bodies hitherto looked upon as almost beyond their means, the first volume is apt to be somewhat antiquated before the last appears. We hope that the publication of this book will be a stimulus to the more rapid production of our British manual. Dr. Prescott's book is a distinct compilation (although an able one), and we look in vain for the amount of original research by the author himself that characterises Mr. Allen's work. On the other hand all the sources drawn upon are very plainly stated and full references are given to the original papers quoted. Having thus put our readers in possession of the relative styles of the two works, we must leave the issue in the hands of the buying public, only repeating that if our British author would only make the same speed, there would be very little room for this work in the home market. The very good remarks in the preface on the subject of the extension of this branch of chemistry (so interesting to us as public analysts) are worth reproducing:—"Respecting the assumed peculiarities of organic analysis, it more and more appears that the differences

between inorganic and organic analysis have been greatly overstated, just as, at earlier periods, the distinction between inorganic and organic chemistry in general was overdrawn. With nearer acquaintance it is seen that the limits of error in determination of carbon compounds are by no means always wider than those in the analysis of metallic bodies. Organic analysis, as the determination of the unbroken compounds of carbon, no longer has an uncertain place in chemical learning."

#### LAW NOTES. TO OUR READERS.

*Some years ago it was decided to discontinue the reporting of ordinary police-court proceedings, and only to give such cases as authoritatively established some point in connection with the working of the acts in which public analysts are interested. After a fair trial of this system, a majority of the members of our Society have expressed a wish that our old practice should be, to some extent, returned to, and, in deference to such request, we have decided to resume the reporting of police proceedings. The line will be drawn at accounts of ordinary cases and reports will only be inserted when any novel, legal, or chemical point arises, or where the certificate of the analyst is in any way attacked. Any member or subscriber connected with such a case, is therefore invited to furnish us with a report of the proceedings signed with his name, not for publication but as a guarantee of exactitude.*

#### GELATINE *versus* ISINGLASS.

On the 13th March, Mr. Latimer Pfander Swinborne was summoned before the Lord Mayor for an infringement of the provisions of the Merchandise Marks Act of 1887, by having applied to certain goods—namely, gelatine or other substance—a false trade description as to the material of which such goods were composed, by which description the goods were falsely described, stated, or indicated to be isinglass. The defendant pleaded "Not guilty." Mr. Besley and Mr. Gray appeared for the complainants; Mr. Poland was counsel for the defence. Mr. Besley, in opening the case, said this was a proceeding instituted by Messrs. Gridley and Co., isinglass merchants, of 4, Bishopsgate Avenue. On February 6th twelve packets of the goods in question were sold at the defendant's premises in St. Andrew's Hill, Queen Victoria Street, the label on the packets stating "by royal letters patent." The learned counsel pointed out that no patent existed after the lapse of fourteen years, and as far as he was instructed the only patent which would warrant that statement was one granted provisionally in the year 1847, and completely registered in 1848. He was not aware of any subsequent patent, but as far as the evidence was before him there had been no patent since that time. That patent had reference to the manufacture of gelatine, and isinglass was not mentioned in it, but only an isinglass cutter. The goods in question were described on the labels as "Swinborne's patent refined isinglass," and it was asserted on the part of the prosecution that this was a false description of the contents. It was alleged in the summonses that it was not isinglass, but gelatine, and that there was not a patent existing which would cover anything like the description. Isinglass was manufactured from



swimming-bladders of sturgeon and other fish. Upon an analysis of a packet of the in question not a particle of isinglass was found, but it was gelatine. Isinglass was marked to a certain extent by non-solubility, and gelatine by solubility. Gelatine can be made from hides or undressed skins. Professor Attfield deposed that he had one of the packets in question. In his opinion the substance was not isinglass but ordinary soluble gelatine. Among other differences, the insolubility in warm water of isinglass as contrasted with the solubility of ordinary gelatine was considered by him as evidence that the material was ordinary soluble gelatine and not isinglass. The witness was examined at length by Mr. Poland as to the composition of isinglass and gelatine. He admitted that the word "patent" indicated that it was not isinglass, but was being a natural substance. Isinglass was chemically one form of gelatine. Dr. Williams of Westminster Hospital, also gave evidence to a similar effect. It was stated when the summonses were served upon the defendant he read them and said, "This was rushed out in 1851." Mr. Poland, at the conclusion of the evidence, submitted his case had been made out by the prosecution. The Lord Mayor, interposing, said he did not think it would be necessary to hear the learned counsel. He did not think that the prosecution had made out their case, nor did he think it one that came within the letter or spirit of the Act. The word "isinglass," by the evidence, was evidence of a term which had been used as applied to gelatinous matters, and it was admitted by authorities that isinglass was the purest form of gelatine. Professor Attfield, in his evidence, had admitted that the word "patent" on the packet proved that it could not be isinglass, which was a natural substance, and, if so, they could no more have isinglass than they could have a patent pear or a patent apple. Mr. Besley said he would like to have the question of law reserved for the opinion of the High Court. Mr. Poland said that ever since 1847 they had described this article in this way. In his evidence the validity of the patent was called in question, and it was then described to be a valuable patent. In 1853 Mr. Swinborne took proceedings against some others, and the Master of the Rolls then said that the plaintiff, Mr. Swinborne, was entitled to a patent for the manufacture of isinglass. They had always described it in this way. The Lord Mayor observed that they could not have a stronger proof that it was right than the fact that a judge had described it as isinglass. Therefore the use of the word as a general term was perfectly correct. The summonses were accordingly issued.

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HOW THEY TREAT "MARGARINE" SELLERS ABROAD.—"A butter merchant in Lisieux in Normandy was charged, in the early part of February, with having sold as butter, mixtures according to analysis between 25 and 40 per cent. of margarine. It was sold under the name of 'Normandy,' 'unequalled,' or 'choice butter.' He was sentenced to three months imprisonment, a fine of 3000 francs and full costs. 20,000 pounds of the butter were confiscated. The sentence to be published in twenty named papers, and to be posted on the doors of the town-halls and market-places of three towns on consecutive market days."

HOW A HAWKER OF MARGARINE CAN BE DEALT WITH IN BRITAIN.—A very interesting case has been decided at the Assizes in Essex, which may be thus summarised. A private person prosecuted a hawker, and had him committed for trial for obtaining money under false pretences, by selling to the said prosecutor margarine as butter. The prisoner was found guilty and sentenced to six months imprisonment, and the Judge told him that if he did it again he would send him to penal servitude. We are endeavouring to get an authoritative report of this trial, and will, if successful, publish the same in an early number.

MR. BURRELL (Cork) sends us the full report of a case (*O'Connel v. Kemp*), where the magistrates have fined a man £2 for selling what was admitted to be a mixture of 25 per cent. of coffee with 75 per cent. of chicory, although contained in a tin with a declaratory label. The *gravamen* of the decision lies in the fact that, in the view of the Court, the label was fraudulent, because while the words "East India pure coffee" were in bright and distinct letters, those following, namely, "specially blended with cultivated chicory" were in very dull letters which scarcely any one could read. An appeal was intimated, the result of which (if persevered in) will be awaited with interest.

## CORRESPONDENCE.

*To the Editor of the ANALYST.*

SIR,—I enclose herewith a copy of my report on the Ottawa water supply, made to the Department of Agriculture.

The source of the water supply is the Ottawa River, the water of which is not subjected to any filtering process before entering the city mains.

Last summer there was an exceedingly and exceptionally small rainfall, and to this may be ascribed, I believe, the presence of the large quantity of dissolved vegetable matter now found in the water. An analysis of this water, made in 1881, by Dr. Baker Edwards (Montreal) showed it to be an exceptionally pure water.

Among the sources of Canadian water supplies we may notice three more particularly: (1) That of the large lakes, *e.g.*, Lakes Ontario, Huron, Michigan, and Superior, to which, apparently, the English standards are applicable; (2) That of the rivers, *e.g.*, Ottawa, which flow through densely wooded districts, and which it would not be wise to judge of with rigour by the English standards, as they necessarily contain more dissolved organic matter of a vegetable origin; and (3) That of springs, the water of which differs greatly from either of the preceding, both as to organic and mineral constituents.

The waters of the second class above named will be very apt to vary in quality according to the season of the year, and I have pointed out the necessity of a systematic and periodical examination, in order to ascertain, if possible, the normal condition of this water.

As a word of *raison d'être* for the late examination I would say that the City of Ottawa has, during the last three months, been visited by a severe epidemic of typhoid fever, for the cause of which many theories have been promulgated, some ascribing it to the water and others to defective drains.

The good work which the Society of Public Analysts inaugurated in England for obtaining greater uniformity in methods of water analysis, and consequently, for the interpretation thereof, has already started here, and valuable data have lately been brought out by Dr. W. Hodgson Ellis, of Toronto, and other analysts, with a view of ascertaining to what extent the English standards are applicable here.—Yours, etc.,

Ottawa, February 27th, 1888.

FRANK T. SHUTE, M.A., F.C.S.,  
Chemist, Dominion Experimental Farms.

[Mr. Shute's report is too long for insertion at present, *in extenso*, but the following are the figures of the analyses referred to.—ED. ANALYST.]

	A	B	C	D
		GRAINS PER GALLON.		
Colour in 2-ft. tube ... ..		Dark Yellow		
Smell at 100° F. ... ..		Slightly peaty		
Chlorine ... ..	·035	·035	·035	·035
Phosphoric acid ... ..		None		
Nitrogen in nitrates and nitrites ... ..	·0080	·0103	·0126	·0109
Free ammonia... ..	·0014	·0014	·0007	·0007
Albuminoid ammonia ... ..	·0091	·0084	·0084	·0084
Oxygen absorbed in 15 minutes ... ..	·1912	·1610	·1708	·1629
Oxygen absorbed in 4 hours ... ..	·3519	·3507	·3507	·3507
Solids ... ..	3·80	3·70	3·92	3·92
Hardness as CaCO ... ..	1·64	1·40	1·55	1·55



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### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS. PEPPER ADULTERATION AND PEPPER ANALYSIS.

By F. M. RIMMINGTON, F.C.S.

(*Read at the Meeting, March, 1888.*)

It has been suggested to me that the subject I have undertaken to bring to your notice this evening would be interesting to the members of the Society, and as very little has hitherto appeared in print on the subject, I thought it possible to throw a little light on a very puzzling question, and hope I may do so.

I don't know what may have been the experience of other analysts, but I have found ground pepper one of the most difficult tasks within the limits of the Adulteration Act, and for this special reason the article is more adulterated than any other. Indeed, so general is the practice, and so varied the system of sophistication, that I have sometimes asked, What is pepper? In the early days of the Adulteration Act the substances used for this purpose were pea-meal, ground rice, flour and mustard husks; but these were soon found to be too simple, and easily discovered, and therefore were replaced by more artful practices. Poivrette has had a brilliant career, and I believe long pepper has had an extensive run. I am not sure that we know all the kinds of pepper that are imported. I have one sample I never saw before in my life, and I have not met with any one able to tell me its history. The number generally mentioned by writers is about six or eight.

I deem it unnecessary to say anything about the history of peppers, but to proceed at once to the method I have pursued in the inquiry, and best suited for detecting the adulteration of the articles. My method is entirely practical, arrived at only after a good deal of experience. I am convinced no purely chemical process can be relied upon,

and the microscope alone is all that is required to detect any addition, except for mineral matter; at the same time it is the most direct method. The difficulty, however, is to estimate percentages. But even with this wonderful instrument, "Seeing is not always believing." What is seen often requires interpretation. Very often minute differences of form and size have to be distinguished, and the manipulation and management of the instrument tends greatly to the success of an examination. If an analyst about to test a sample of pepper puts a little on a slide with a drop of water, and then examines it with the microscope, the probability is he would observe nothing wrong, assuming that no gross fraud is practised, such as cereal-starches dhuriv, etc. But if finely-ground rice, or poivrette, or long pepper, or pepper husk was used, this adulteration would not be detected. But



FIG. 1.

if he introduce one or two grammes of the powder into a test-tube one-half or four-fifths of an inch wide, and then half fill the tube with alcohol, and shake it well, and let it stand about half a minute to settle, and pour off the spirit into a small beaker, then repeat the process, and again pour the spirit into the beaker as close as possible without disturbing the deposit, when the deposit in the tube is now treated in the same way with water, it is rendered milky by the presence of resinous matter; this may be removed by the addition of two or three drops of a solution of potash, or by pouring off the milky fluid, and adding more water. The second addition of water need not be poured off. The tube is now allowed to stand for five or ten minutes, and the deposit will be found to have arranged itself in two or three strata; the upper stratum (if a little be withdrawn with a pipette) will be found to contain the elements of most



interest. A very small portion of this matter is now to be placed on a slide, and examined with the microscope, with a power of 150-250 diameters, and a sub-stage condenser; every particle should be seen clear and well-defined, and any foreign bodies will be recognised. If the starch granules are to be examined as to form and size, a magnifying power of 250 or more will be necessary. The sediment in the beaker may also be examined for what may happen to be there.

One of the chief reasons of my presence to-night is to submit to your inspection certain specimens, and to obtain your opinion upon them as to their nature and origin. During the past year I have had many samples of pepper brought me by the inspectors and dealers. Nearly all of them presented one singular feature, which puzzled me much



FIG. 2.

to explain; it was quite evident the samples contained something beyond the ordinary kind of pepper. The only conclusion I could come to was that it was long pepper. On making some sections of this, I found a stratum of cells lining the inside of the little berries (for each of the little spherules is a separate fruit) with a row of large elongated cells filled with spherical bodies, so closely resembling the clusters of similar bodies (see Fig. 5) I found in the peppers, that I could distinguish no difference (see Fig. 1). I therefore have come to the conclusion that these peppers are fraudulent admixtures with long pepper. But there are two things that tend to throw a doubt upon this point. Ground long pepper is a dark and dirty-looking powder, and if added to ground white pepper would spoil its appearance by lowering its colour, and thus destroy the advantage sought for. It occurred to me whether there was any method by which

the dark-coloured cellular tissue which forms the structure of the spike, could be removed after grinding, as by sifting?

My past experience almost leads me to the conclusion that there are several substances used for mixing with pepper, but without some previous knowledge of what they are, it would be impossible to identify them.

*Cellular structures.*—The cellular structure of pepper is pretty clearly marked, but it varies in some respects in different kinds, and the eye of the microscopist requires a good deal of training to enable him to discern readily the different forms. The cells of common black and white pepper have generally sharp acute angles (see Fig. 2), and the figure approaches a square, but this varies considerably, and it is not possible to give

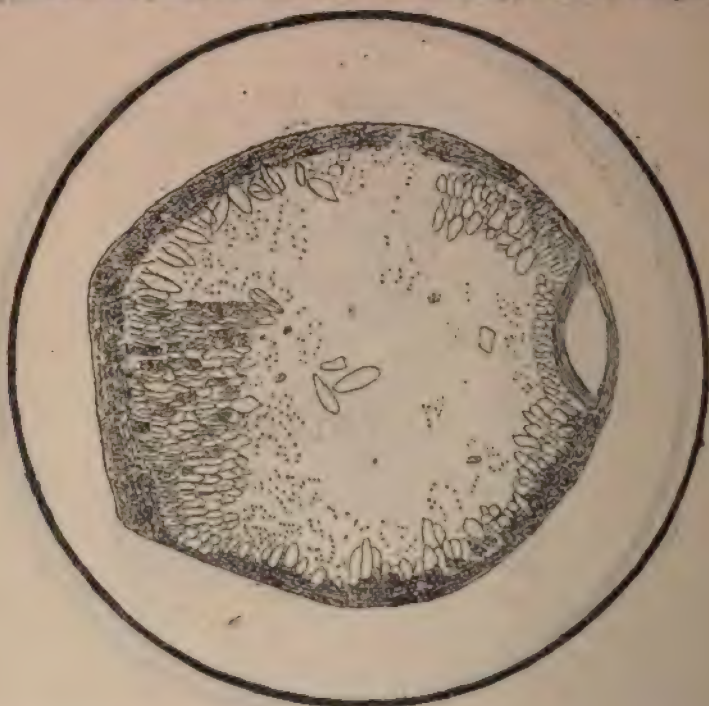


FIG. 3.

a clear and correct definition. The starch granules are quite round, and possibly spherical, and very small.

The structure of long pepper is also well marked. The fruit-spike consists of a mass of fibrous and cellular tissues in which is imbedded a great number of small baccate fruits—it is these little berries that constitute the useful and active part of the fruit, the investing structures of the spike being useless. When a section (Figs. 3-4) of a spike of this pepper is examined microscopically the interior of these little berries is found lined with a stratum of cells immediately beneath the rind or cortex of an elongated and somewhat cylindrical form, and truncated ends; the cells are comparatively large, and are filled with spherical bodies that have the appearance of starch cells. But besides these bodies there is another form of starch not enclosed in cells, but apparently imbedded, and intermixed amongst the large cells. This is the characteristic starch of long pepper;



is much larger than the starch granules of black or white pepper, and has a well-defined angular figure; it almost approaches the size of rice starch.

It is the close resemblance of the spherical bodies contained in the large cells, to the conglomerate masses (see Fig. 5) observed in almost every sample of pepper I have examined, that convinces me they are the result of a fraudulent admixture, and I entertain the hope that by drawing attention to the matter I shall get the opinion of other analysts.

The next point I have to submit to the meeting is whether the mixture of long pepper with white is to be regarded as a fraud under the Food and Drugs Act. That is a commercial fraud there is no doubt. Or why use it? The injury to the con-



FIG. 4.

sumer may not be serious, but the injury to honest dealers is very great. Long pepper is inferior to white pepper both in strength and flavour.

#### LITERARY NOTICES.

Several papers appear in the *Ph. Journal* for 1874, on pepper adulteration.

A paper by Bourchardat on "Adulteration of Pepper" appears in the same journal. He gives no analytical process, but enumerates the many substances used for the purpose, such as dried potatoes, chalk, lentil flour, and poivre léger and poivre blanc, all of which may be detected microscopically. He says the powder of pepper is characterised by its starch. The grains are of various forms and dimensions.

Dr. Wynter Blyth also gives several analyses—he also enumerates liquorice, the bones from olives, bone dust, and salt as adulterants. His analyses go to show that long pepper is very inferior to other kinds as regards strength.

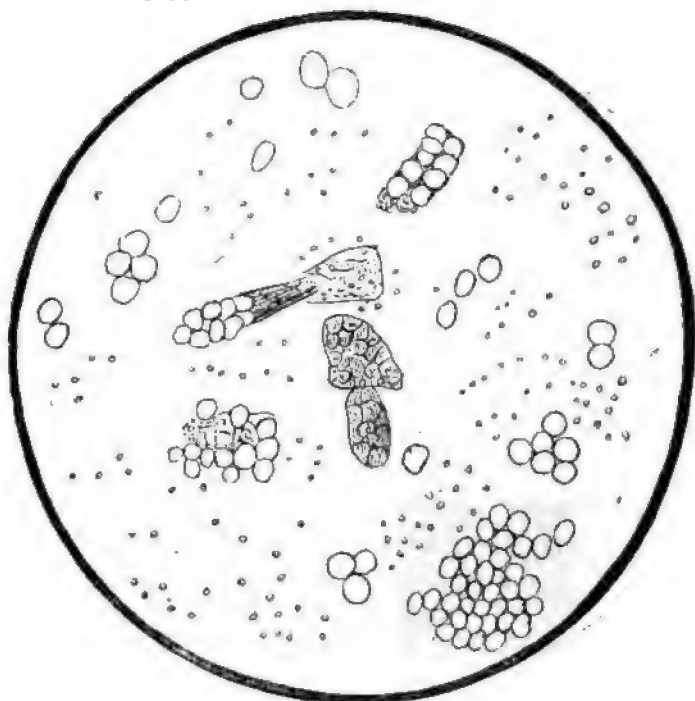


FIG. 5.

Evans, *Ph. Journal*, 1860, chiefly on the structure and the use of mustard husk, capsicum, and cereal starches as adulterants. Several prosecutions for pepper adulteration in this volume, some good plates in the next volume.

Battershall, in his recent work on "Food Adulteration," says that out of 386 samples of pepper examined by the chemists of New York and Massachusetts, 236, or 61 per cent., were found to be adulterated.

#### DESCRIPTION OF THE PLATES, TAKEN FROM DRAWINGS.

Fig. 1 is a characteristic section of long pepper, showing the large spherical bodies mentioned in the text.

Fig. 2 is a section of white pepper, the dark spots in the cellular tissue are resinous matter, containing crystals of peperin, etc.

Figs. 3-4 are other sections of long pepper, showing starch grains and cellular structure.

Fig. 5 represents the large starchy bodies as separated from other matters in adulterated samples of pepper, magnified 250.

The plates 1, 3, 4, being reduced copies of original drawings, make all the structures too small as seen with a  $\frac{1}{4}$  objective, magnifying 150 diameters.

#### SOME ANALYSES OF YEAST.

By ROWLAND WILLIAMS, F.I.C., F.C.S.

(Read at Meeting, March, 1888.)

DURING the last few months I have had occasion to determine the percentages of moisture, ash, and starch in several samples of yeast which have been submitted to me for analysis.



As I can find no record of similar estimations in the ANALYST or other chemical journals, I thought my results might be of some little interest to members of our Society, and it is with that intention I now publish them.

All the samples were carefully examined for starch, but that substance was only found in three out of the ten samples, and in one case in such minute amount that its presence must be attributed to accident. In the other two samples, which contained considerable proportions of starch, it was estimated according to the plan of decantation suggested by Mr. W. F. Stock in the ANALYST for May, 1887.

On reference to the table of results, it will be seen that the amount of mineral matter in the different samples varies between about  $1\frac{1}{2}$  and 3 per cent., the average percentage being 2.22.

The proportion of water in the pure yeasts is fairly constant, most of the percentages lying between 70 and 75, the average being 72.94. The addition of a large amount of starch reduces the percentage of moisture very considerably. The moisture determinations were made in the usual way by thorough drying in the water bath, and the estimations of ash by ignition in platinum crucibles. All the samples were slightly acid, whilst the mineral matter was in each case alkaline in reaction.

	Moisture.	Ash	Organic matter.	Starch.
No. 1 mixed .. ..	60.84	1.54	37.62	21.2
No. 2 " .. ..	67.19	2.06	30.75	12.3
No. 3 pure .. ..	71.50	2.43	26.07	trace
No. 4 " .. ..	76.69	3.13	20.18	absent
No. 5 " .. ..	70.27	2.01	27.72	"
No. 6 " .. ..	74.87	2.17	22.96	"
No. 7 " .. ..	74.37	2.02	23.61	"
No. 8 " .. ..	70.02	2.29	27.69	"
No. 9 " .. ..	71.79	2.37	25.84	"
No. 10 " .. ..	74.03	2.25	23.72	"

#### SOME ANALYSES OF GOOSE FAT.

By W. C. YOUNG, F.C.S., ETC.

(Read at Meeting, March, 1888.)

HAVING had a sample of goose fat submitted to me to ascertain whether it was genuine or not, and finding, on reference to the text-books available to my hand, that the only mention of goose fat occurred in Allen's "Commercial Organic Analysis," and that merely consisting of the saponification equivalent, I was led to make an examination of a few authentic samples of goose fat. These results being somewhat interesting, I thought they would be acceptable to the Society, especially as so little appears to be known on the subject.

#### RESULT OF EXAMINATION OF FOUR SAMPLES OF GOOSE FAT.

	A.	B.	C.	D.
Specific gravity at 100° F. (water at 100° F. = 1000)	908.9	909	909.3	908.3
Insoluble fatty acids (per cent.) .. ..	2.27	3.46	3.1	0.7
Soluble " " " " .. ..	93.70	92.71	92.4	95.7
Saponification equivalent .. ..	284	283	296	304
Per cent. KHO required .. ..	19.70	19.8	18.94	18.4
Schickert's process .. ..	nil	nil	nil	nil

It will be seen by these results that the specific gravity was very high, and that three out of the four contained a notable proportion of soluble fatty acids. It is evident that if such a fat were mixed with butter a considerable quantity might be present without being detected if these data were solely relied upon, fortunately, the soluble fatty acids are not volatile, therefore any considerable admixture of such a fat with butter would be detected by means of Reichert's process.

#### DISCUSSION.

Dr. VIETH said there would be no difficulty in detecting goose fat in butter, without any analysis. The most remarkable statement was that there was no particle of soluble fatty acids volatile. He had never made an analysis of a bird's fat, but had of beef and mutton, fallow deer, stag, and badger, and he found all these fats were within the usual limits; the insoluble fatty acids were within 95 and 96. He had also examined ewes' and goats' milk, and found them very similarly composed, the insoluble fatty acids were 87 or 88, the fat of mare's milk he found to contain not less than 92.6 of insoluble fatty acids, and it was remarkable that after saponification the smell of butyric ether was very faint indeed.

Mr. HEHNER said that elephant's milk and pig's milk had been examined, and found to correspond with that of the cow.

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#### THE IODINE ABSORPTIONS, COMBINING WEIGHTS, AND MELTING-POINTS OF CERTAIN FATTY ACIDS.

BY ROWLAND WILLIAMS, F.I.C., F.C.S.

(*Read at the Meeting, March, 1888.*)

THE iodine absorptions of a large number of oils and fats may be found in "Allen's Commercial Organic Analysis" (Second Edition, Vol. ii., page 50), but, so far as I know, the percentages of iodine absorbed by the fatty acids themselves have never yet been published.

This is a matter of considerable importance, as it is often necessary to examine very minutely the nature of the fatty acids separated during the analysis of soap. The melting-point and combining weight of the fatty acids are usually relied upon to give the required information, and in many cases the data thus obtained are sufficient for the purpose. If, however, any doubt exists, the determination of the iodine absorption of the fatty acids often proves of great value.

I have recently been working very extensively upon the fatty acids from tallow and lard, and from cotton-seed, olive, linseed, rape, castor, cocoa-nut, palm, and sperm oils. Several samples of each kind of oil or fat were examined, and the averages of all the results obtained are recorded in the annexed table. For the sake of comparison I also give the melting-points and combining weights of some of the fatty acids, which appear in "Allen's Organic Analysis" (Second Edition, Vol. ii., pages 213 and 315), most of which agree fairly well with my own results.



Name of Oil or Fat.	Iodine Absorption.	Combining Weight.		Melting Point.	
		R. W.	Other observers.	R. W.	Other observers.
Lard .....	41.3 per cent.	276	270 to 285	119° F.	—
Cotton-Seed Oil.....	64.2 "	296	270 to 285	100° F.	111° F.
Olive Oil .....	115.7 "	289	275 to 337	96° F.	95° F.
Linseed Oil .....	90.2 "	286	270 to 285	81° F.	79° F.
Sesame Oil .....	178.5 "	283	307	75° F.	75° F.
Castor Oil .....	105.6 "	307	321	71° F.	67° F.
Peanut Oil .....	93.9 "	292	290 to 306	—	—
Coconut Oil.....	9.3 "	201	196 to 204	75° F.	75° F.
Palm Oil .....	53.4 "	263	269 to 273	114° F.	122° F.
Peru Oil .....	88.1 "	305	289	56° F.	—

The combining weights are the figures obtained by dividing the number of milligrammes of fatty acids taken for the titration by the number of c.c.'s of normal alkali required.

The melting-points were determined by the ordinary capillary tube method.

An examination of the above figures will show that my combining weights agree very closely on the whole with the results of other chemists. My melting-points also correspond very well with those of other observers, the only important discrepancies being in the case of palm oil and lard.

The low melting-point obtained by me in the case of lard may possibly be due to some of the samples which I examined having been made from the fat of the entire animal, while the sample of which Mr. Allen gives the melting-point, was most probably prepared solely from the omentum. As might naturally be expected, all the iodine absorptions are somewhat higher than those of the oils and fats from which the fatty acids were derived.

These results will, I hope, prove of some benefit to those engaged in the analysis of fats, and in other cases in which an examination of fatty acids is necessary.

*(Conclusion of the Society's Proceedings.)*

#### NOTE ON THE ABSORPTION OF AMMONIA BY ACID SOLUTION IN NITROGEN DETERMINATIONS WITH SODA-LIME.

By I. S. HAYNES.\*

Among the possible sources of error in the determination of nitrogen by the method of Arrhenius and Will is the failure of the ammonia to be completely absorbed by the acid solution, in case the combustion proceeds very rapidly and the ammonia is considerably diluted with other gases. The following determinations were made to test the effect of rapid combustion on the absorption of ammonia, and, incidentally, the danger of small particles of soda-lime being swept past the asbestos plug into the acid solution.

\* American Chemical Journal.

Although undertaken simply as one of the series of exercises in the undergraduate course of study and practice in this laboratory, the results may not be unworthy of record.

The method followed was that described in a previous article as usual in this laboratory, except that the packing of the asbestos plug in the anterior end of the tube and the time of combustion were varied, as shown in the tabular statement herewith. The ammonia was caught in an ordinary Knop and Arendt bulb apparatus (with four bulbs) containing 10 c.c. of a rather concentrated standard solution of sulphuric acid, of which 1 c.c. corresponded to about 10 mgm. of nitrogen. About 0.25 grm. of ammonium sulphate mixed with stearin previously proved to be free from nitrogen, was used for each determination, and furnished ammonia sufficient to neutralise about one half of the acid.

In the determinations numbered 1, 2, and 3 in the table, the anterior asbestos plug was some 2-3 cm. long and packed reasonably tight, as is usually done in our work. In No. 4 it was shorter than usual, and care was taken to pack it very tightly by holding the tube perpendicular with the anterior end down and pressing upward against the asbestos with the flat end of a lead pencil. The soda-lime above furnishes a firm backing, so that the asbestos may be packed as closely as desired. Reversing the tube and tapping it gently, its contents will settle down from the asbestos, leaving a space which can be closed up as much as necessary by pushing the compacted plug toward the soda-lime. In Nos. 5 to 7 the plug was put in loosely, as might be done by a careless operator. The figures in the table for number of bubbles per minute were estimated by countings of the numbers in different minutes, and are of course only approximate. In Nos. 4 and 7 the effort was made to push the combustion as rapidly as could be done without danger of accident. The flow of gas was much more rapid, and the bubbles were much larger than would be the case in any ordinary well-conducted analysis. This will readily be seen when we consider that the quantity of diluting gases, from the decomposition of nearly a third of a grm. of stearin in each case, was large, and that the time of the combustion was only 12 minutes, instead of from 30 to 60 minutes, which the combustion commonly takes. Add that the quantity of acid solution was rather small, only 10 c.c., and it is evident that the conditions were very unfavourable for complete absorption of the ammonia. The percentages of nitrogen in Nos. 1 to 4 average 21.14. This figure accords very closely with those obtained by other gentlemen who made determinations of nitrogen in the same material and at the same time, by the soda-lime and other methods.

*Determinations of Nitrogen in Ammonium Sulphate.*

Number.	Asbestos Plug.	Rapidity of Combustion.	Time of combustion. Minutes.	Number of bubbles per minute. Approximate.	Ammonium Sulphate used. Grms.	Nitrogen found. Per cent.
1	Moderately tight.	Rather slowly.	60	60	0.2457	21.12
2	" "	Moderately fast.	35	70	0.2458	21.15
3	" "	" "	35	70	0.2458	21.15
4	Short and very tight.	Very fast.	12	90	0.1796	21.16
5	Loose.	Rather "	25	70	0.2630	21.49
6	Short and loose.	Very "	13	85	0.2558	21.69
7	Long "	" "	12	90	0.2928	21.37



Comparison of the figures in the last column of the table shows no loss of nitrogen, even in No. 4, in which the flow of gas was so rapid and the bubbles were so large. To still further test whether ammonia escaped absorption, a second bulb tube with acid solution was connected with the first so that the gases which came from the first passed through the acid in the second. Even in No. 4, Nessler's test gave no reaction for ammonia in the second tube. It is clear, then, that despite the considerable dilution and extremely rapid flow of the gases through the 10 c.c. of acid solution, no ammonia escaped absorption. But it is to be remembered that the quantity of ammonia was only equivalent to about half of the acid in the solution.\*

The large figures of "nitrogen found" in Nos. 5, 6, and 7 are evidently due to the fine particles of soda-lime which were carried through the loosely packed asbestos plugs by the rapidly passing gases. The necessity of proper packing of the asbestos plug is too obvious to need dwelling upon.

The principal fact brought out in these experiments is that the ammonia, the quantity of which was considerable, was completely absorbed by the acid solution in the cases where the combustion was completed in as short a time as twelve minutes, and where the flow of gas was far more rapid than would occur in any ordinary work. The inference is that the absorption of the ammonia is more certain, and the danger of loss less, than is frequently supposed.

#### A METHOD FOR THE SEPARATION AND DETERMINATION OF BORIC ACID.†

By H. N. MORSE AND W. M. BURTON.

The indicator, tropaeolin *OO*, not being sensitive to boric, carbonic, and silicic acids, or to their salts, it is practicable to liberate exactly one or all of these acids in any mixture of inorganic substances. In other words, if to a solution containing silicates, carbonates, and borates, and any other inorganic salts or hydroxides, we add tropaeolin and then dilute sulphuric acid, we shall not obtain an acid reaction until all of the silicic, carbonic, and boric acids have been liberated. In the meantime, any oxides or hydroxides which were present will have been converted into sulphates, so that the mixture will finally consist of neutral salts, water, silicic, carbonic, and boric acids. If we now add to this mixture a sufficient quantity of dehydrated copper sulphate, the water will be absorbed and the silicic acid dehydrated. The mass will then consist of neutral salts, silica, and boric acid, of which only the boric acid is soluble in absolute alcohol. These facts enable us to separate boric acid with great exactness from nearly all other forms of inorganic matter. The only classes of salts known to us at present which are at all likely to interfere with such a separation, are the chlorides and the compounds of iron, but both of these may be readily removed before the acids in question are liberated. The boric acid thus separated may be determined as a meta-borate of calcium, magnesium, or barium. We prefer the last form; because a mixture of carbonate and meta-borate of barium, being neither markedly hygroscopic nor capable of absorbing carbon dioxide, can

\* See Gassend and Quantin, *Zeitschr. anal. Chem.*, 1882, 278.

† *American Chemical Journal*.

be brought to a constant weight more readily than one containing the oxide of calcium or magnesium. Moreover, the use of barium enables us to employ a standard solution of hydroxide to receive the alcoholic extract of boric acid, thus saving the labour of bringing the absorbing material itself to a constant weight.

*The Reagents.*

1. An aqueous solution of tropaeolin OO.
2. A standard solution of sulphuric acid of such strength that one cubic centimetre of it is equivalent to twenty milligrammes of barium carbonate.
3. A solution of barium hydroxide approximately equivalent to the sulphuric acid. This is prepared by agitating the solid commercial hydroxide with a little cold water, filtering, and washing with cold water. The residue, thus freed from the caustic alkalis, is transferred to a flask or bottle and treated with water. From this solution the more dilute ones are prepared as they are needed.
4. Dehydrated copper sulphate prepared from the pure salt by heating in an air-bath at  $150^{\circ}$  until the whole mass assumes a uniform light colour. It is necessary to use pure copper sulphate for this purpose. The presence either of chlorides or of iron salts is not permissible, because of the solubility of cupric chloride and ferric sulphate in alcohol. It is not advisable to attempt to remove more than four of the five molecules of water of crystallisation, because of the danger of decomposing some of the salt at high temperatures. Such decomposition would result in the formation of free sulphuric acid, which would be extracted along with the boric acid by alcohol.
5. Absolute alcohol prepared by digesting for two or three days with dehydrated copper sulphate an alcohol which has previously been distilled over lime. The complete drying of the alcohol is important.

*The Mode of Procedure.*

If the boric acid is in solution, the liquid containing it is made slightly alkaline with caustic potash, and evaporated on a water-bath in a porcelain dish having a width of about 100 millimetres. The volume of the solution should be reduced to 10 or 12 cubic centimetres. The separation of salts during evaporation will not interfere with subsequent operations. If the material containing the boric acid is not soluble in water, a silicate, for example, a solution is effected by the method recommended by one of us for the decomposition of chrome iron.\* About four grammes of the purest solid potassium hydroxide are placed in a nickel crucible† and heated with a small flame until the fused mass becomes perfectly tranquil. The crucible is allowed to cool and the finely ground and weighed material spread over the surface of the solidified hydroxide. The whole is then heated for two hours to a temperature just sufficient to keep the hydroxide in a fused condition. During the decomposition the mass is frequently stirred with a stout platinum wire, which is allowed to remain in the crucible. This method of bringing the acid into a soluble condition is to be preferred to fusion with the alkaline carbonates, since it yields a mass from which everything soluble can be readily extracted by water. The crucible is placed on its side in a porcelain dish, treated with hot

\* *American Chemical Journal*, iii., 163.

† A silver crucible is not to be recommended, because of the solubility of silver in potassium hydroxide.



water, and the soluble separated from the insoluble matter by filtration and washing. The filtrate is then evaporated to the small volume previously mentioned. If the material thus decomposed contains iron, the contents of the crucible must be treated with not less than 400 cubic centimetres of water, and the dish in which the solution is made should be heated on the water-bath for a considerable time, with replacement from time to time of the evaporated water. Otherwise the separation of the iron may not be complete.

The solution of borate is treated, under proper covering to prevent loss by spattering, with a drop or two of tropaeolin solution, and then with the ordinary desk solution of sulphuric acid until a distinct and permanent acid reaction is obtained. The material on the under side of the cover is washed back into the dish with the least possible amount of water, and the slight excess of sulphuric acid carefully neutralised by means of very dilute caustic potash. The solution of boric acid, which should not exceed 20 cubic centimetres in volume, is now thoroughly dried by slowly stirring into it the anhydrous copper sulphate, care being taken not to permit any considerable rise in temperature. The mass is afterwards pulverised with a pestle.

The extraction apparatus consists of a narrow Erlenmeyer flask having a capacity of about 150 cubic centimetres, and a straight chloride of calcium tube somewhat larger than is necessary to contain the mixture which is to be extracted. The narrow portion of the tube is plugged with cotton wool. To its lower end is attached, by means of rubber tubing, a small glass tube which reaches to the bottom of the flask when the stopper carrying the extraction tube is in position. The quantity of the dilute barium hydroxide solution which is equivalent to 25 cubic centimetres of the standard sulphuric acid, is run into the flask and the apparatus attached to the filter pump. This quantity of the hydroxide would weigh, if converted into carbonate, just 500 milligrammes. During the filling of the extraction tube with the copper sulphate mixture, the outside is occasionally tapped to render the material more compact.

The porcelain dish is washed with several small portions of absolute alcohol, which are then poured into the tube. When the whole of the copper sulphate has become wet with the alcohol the pump is shut off and the mass allowed to soak for fifteen minutes. Afterwards five other portions of absolute alcohol, of 15 cubic centimetres each, are poured into and slowly drawn through the tube, care being taken to allow each portion to filter through completely before another is added.

Finally, the excess of the barium hydroxide is precipitated by passing into it a current of carbon dioxide; the contents of the flask are transferred to a weighed platinum dish, evaporated to dryness, and heated to constant weight over a triple burner.

The quantity of boric anhydride is found by the following proportion:—

Mol. weight of  $B_2O_3$ —mol. weight of  $CO_2$ : mol. weight of  $B_2O_3$ : : weight found—  
theoretical weight of the barium as carbonate: weight of  $B_2O_3$  found.

If the atomic weights of Clarke have been used throughout, the difference between the weight found and the calculated weight of the carbonate (500 milligrammes) is to be multiplied by the number 2.697012; if those of Meyer and Seubert have been employed, this difference is to be multiplied by the number 2.701822.

The accuracy of the method was tested by determining the boric acid in pure borax

glass (prepared by ourselves), in datholite from Bergen Hill, and in tourmaline from Pierrepont, N. Y.

We give the results below:—

#### I. *Borax Glass.*

Measured portions of a standard solution were employed.

Theoretical percentage of  $B_2O_3$ , 34.61. Found, 34.64, 34.58, 34.64, 34.57, 34.57.

Five determinations made by Mr. R. L. Henderson of this laboratory gave the following percentages of  $B_2O_3$  in our borax glass, 34.60, 34.60, 34.59, 34.58, 34.59.

#### II. *Datholite from Bergen Hill.*

Theoretical percentage of  $B_2O_3$ , 21.82. Found, 21.75, 21.68, 21.62, 21.65, 21.64. Mean, 21.67.

#### III. *Tourmaline from Pierrepont.*

This mineral was recently carefully analysed by Dr. Riggs.

Five determinations of the boric acid were made by ourselves, one by Mr. C. C. Blackshear and one by Mr. Henderson.

Percentage of  $B_2O_3$  found by Dr. Riggs, 10.15, 10.00, 10.31; by ourselves, 10.03, 10.08, 10.11, 10.03, 10.13. Mean, 10.08. Found by Mr. Blackshear, 10.02; by Mr. Henderson, 10.12.

### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ANALYSIS OF ORES OF AURIFEROUS TELLURIUM. F. M. HORN. *Zeitschr. f. angew. Chemie.*, No. 6.—The analysis is easily performed by gently heating the finely powdered ore in a current of dry chlorine (Berzelius' method). The author succeeded equally well by treating the mineral with hydrochloric acid, with addition of a little nitric acid. The solution must be evaporated to dryness and the residue taken up with dilute hydrochloric acid, to which a little tartaric acid has been added. Any insoluble residue consists of silica, argentic chloride, and plumbic sulphate, and must be assayed for silver by the usual process. The solution is freed from nearly all the lead, by means of dilute sulphuric acid, and then heated with slight excess of ferrous sulphate, which will cause a separation of metallic gold, which can be collected and weighed. From the filtrate the tellurium may now be precipitated as metal by adding large excess of sulphurous acid, and heating for some time to 80° C. Antimony, if present, may now be estimated by expelling the sulphurous acid and passing hydrogen sulphide, which will precipitate the antimony as sulphide. This is finally converted into oxide by treatment with fuming nitric acid, and weighed.

If the ore contains no gold or antimony, but copper or arsenic, the process starts just the same, there being, of course, no need for ferrous sulphate. After the tellurium has been separated, and the excess of sulphurous acid expelled, the copper and arsenic may be jointly precipitated by hydrogen sulphide.

After fully oxidising the washed precipitate with nitric acid, the copper may be readily separated from the arsenic by means of potassic hydrate. The arsenic may be estimated by neutralising the solution with nitric acid and then adding excess of ammonia and magnesia mixture, etc.

Besides silver, gold, arsenic, antimony, tellurium, copper, lead, there are generally small quantities of iron, zinc, lime, magnesia, sulphur and sulphuric acid, which are determined by the usual methods.

L. DE K.



ESTIMATION OF PARAFFIN IN FATS, ETC. R. ZALOZIECKI. *Dingl* 267, p. 274.—Analysts who have had experience of the fairly accurate but troublesome sulphuric acid process, will no doubt welcome the author's method, which, however, does not work with mixtures of paraffin and beeswax. It is based on the practical insolubility of paraffin in a mixture of alcohol and fusel oil. Ten grammes of the sample (fats, fatty acids, resin, rosin-oil) are treated with fifty grammes of fusel oil, and then mixed with fifty grammes of alcohol. After standing for a few hours, the precipitated paraffin is filtered off, and washed with a mixture of two parts of fusel oil and one part of alcohol. The paraffin is now dissolved in benzol, and this solution evaporated in a weighed dish, and the residue dried at 125° C. and finally weighed.

L. DE K.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

METHODS FOR DEFINITELY PROVING THE PRESENCE OF COTTON OIL OR OF OIL OF SESAME IN OLIVE OIL. M. MILLIAU. *Arch. de Pharm.*, 3, 161.—The author has applied Becchi's test (*ANALYST*, Vol. xii., p. 170), based upon the reducing action of cotton oil upon argentic nitrate, but he finds that some varieties of the pure olive oil give a slight reduction, owing to certain causes which he points out. If, however, the fatty acids be first separated and the test be then applied to them, the acids from pure olive oil give no reduction, while those from cotton oil always reduce the silver. On this assumption he has based the following process:—Place 15 c.c. of the oil into a porcelain basin and warm it up to 110° C. Make a mixture of 15 c.c. of a solution of sodium hydrate (40 degrees Baumé) with 15 c.c. of alcohol of 92 degrees, and add this solution very gradually to the heated oil, taking care that the temperature is maintained during the process. When a homogeneous mass has been obtained, distilled water is added drop by drop, so as not to cool the paste or to form clots, and this is continued until 500 c.c. of water have been introduced. The whole having been boiled for a few minutes, dilute sulphuric acid (1 in 10) is added, so as to produce a slight excess of acidity and the fatty acids are thus obtained floating on the surface. The author does not seem to consider it necessary to wash these acids, but he simply removes about 5 c.c. by means of a silver spoon, and puts this amount of the separated acids into a test tube with 20 c.c. of alcohol, and warms until dissolved. To this liquid he adds 2 c.c. of a 30 per cent. solution of argentic nitrate, and heats the whole on the water bath until about one third of the fluid is evaporated off, when the test is complete. If the olive oil be pure, the fatty acids remain unaltered, but if cotton oil be present the acids float to the surface as a black paste. By this method the author has easily detected a one per cent. adulteration.

*For Sesame Oil.*—To recognise the presence of this oil in olive oil, the author finds that Baudouin's reagent is the best, but here also a pure oil may be unjustly condemned when working on the oil itself. If, however, the separation of the fatty acids be first carried out, the results are accurate. In this case, after saponification and separation of the acids, they must be perfectly dried at 110° C. They are then put into a tube and shaken up with Baudouin's reagent (hydrochloric acid and sugar), when the characteristic red colour will be produced.

W. H. D.

In the last issue of the "International Review of Food Adulteration," published in Holland, an English firm are charged with offering for sale to manufacturers of chocolate a brown substance designed to improve the colour of their manufactures. The powder is stated to be specially recommended as "quite non-injurious, and being moreover very light, and of a great staining power." This powder has been found to be a mixture of the oxides of iron and aluminium. We should fancy that foreign makers of chocolate did not require any instruction from England as to the use of red ochre. W. H. D.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

CHARACTERS AND REACTIONS OF STROPHANTHIN. M. CATILLON. *Arch. de Pharm.*, 3, 102.

—The author has prepared a large quantity of this body, and he finds it to be a crystalline substance separating from its solution in minute needles, which are transparent when wet, but opaque when dried. Its solution is neutral to test paper and gives no precipitate with the ordinary reactions for alkaloids, but gives with excess of tannin a copious white precipitate. Sulphuric acid causes a greenish precipitate becoming black on standing, but to obtain this result rapidly the liquid should be gently warmed. When the acid is applied directly to a crystal of the poison, it develops a fine emerald-green, passing through reddish-brown to black. With hydrochloric acid, a reaction is produced likely to cause a confusion with that for digitalin, but while this latter substance gives with ferric chloride in the presence of sulphuric acid a blue colouration, strophanthin produces a green colouration under similar circumstances. With nitric acid it develops a pale rose colour, becoming orange-yellow on gently warming. These reactions are, however, only strictly characteristic of the article derived from the strophanthus Kombe, because the author has obtained other varieties of the drug, which were not so derived, and which failed to give all the above-mentioned tests. W. H. D.

ESTIMATION OF GLYCERIN. A. JOLLE. *Zeits. Nahrung.*, i., 221.—In applying the oxidation process by alkaline permanganate, and subsequent estimation of the oxalic acid formed, to the estimation of the glycerin produced by the saponification of a fat by alcoholic potash, an error is introduced by the oxidation of a portion of the ethylic alcohol used to the same acid. The author finds that this error is entirely avoided by the use of a cold solution of an alkaline manganate instead of the heated alkaline permanganate. By the author's modification, 3 grammes of the fat are saponified by alcoholic potash, the alcohol is evaporated off and the acids are thrown up by hydrochloric acid. They are then filtered off in the usual way, washed, and the glycerin is estimated in the filtrate. This filtrate, which should be diluted to 200 c.c., and mixed with 5 grammes of potassium hydrate, is treated at ordinary temperature, with a sufficiency of a solution of 4 grammes of potassium manganate and 5 grammes of potassium hydrate in a litre of water. The whole should remain slightly green at the end of the reaction, and this slight colour should then be discharged by a little sulphuric acid. Finally the oxalic acid produced is estimated with calcium chloride in the usual manner.

W. H. D.



ESTIMATION OF NICOTINE. J. BIEL. *Pharm. Zeits. f. Russ. and Drug. Circ.*—We possess a copious literature on the estimation of nicotine in tobacco leaves, cigars, etc. Schlossing exhausts the powdered leaves (previously moistened with ammonia) in an extraction apparatus with ether, subjects the ethereal solution to distillation till all the ammonia has been removed, lets the remainder of the ether evaporate spontaneously, and titrates the residue with normal sulphuric acid, of which 1,000 c.c. are equal to 162 grams. of nicotine.

This method yields too high results, because some of the ammonia remains with the nicotine, and 17 parts of ammonia require for titration as much sulphuric acid as 162 of nicotine.

Wittstein does not use ammonia and ether, but water acidulated with sulphuric acid. The extract, after being neutralised with an alkali, is subjected to distillation, and the distillate titrated with normal sulphuric acid. The sulphate of ammonia formed is titrated separately by evaporating, extracting the residue with strong alcohol, and in the insoluble residue estimating the sulphuric acid with baryta. By calculation the sulphate of ammonia is found and deducted from the total weight.

Kosutary extracts the tobacco leaves (previously mixed with milk of lime) with water, shakes the extract with benzol (petroleum ether), and fixes the nicotine with titrated sulphuric acid, by repeatedly shaking the benzol extract with the acid. Excess of the latter is re-titrated.

Dragendorf employs Mayer's reagent for estimating nicotine in the aqueous extract.

Skalweit boils the finely-powdered tobacco (previously moistened with sulphuric acid) with alcohol for two hours, using a backflow-cooler; then distils off most of the alcohol from 100 c.c., pours in diluted solution of potash through a funnel, and distils from a sand-bath till the drops cease to react on red litmus paper; then titrates with normal sulphuric acid.

Kissling\* moistens the finely powdered tobacco with alcoholic solution of soda, extracts after twenty-four hours, with ether in Soxhlet's extraction apparatus, distils off the ether, adds to the residue diluted solution of soda, and finally distils off the nicotine in a current of live steam, and titrates with sulphuric acid.

In contrast to all these chemists, Hager does not estimate the isolated nicotine volumetrically, but as hydrochlorate by allowing the nicotine to remain for several days under a bell glass together with hydrochloric acid. The hydrochlorate is very hygroscopic, and must be dried for some time over sulphuric acid. Dragendorf has shown, however, that even with this precaution the obtained weight will be greater than the theoretical, and that the hydrochlorate probably still contains two molecules of water of crystallisation.

Kissling has devised a method by which the nicotine can be estimated directly from the commercial extract. The extract is diluted with water, solution of soda added, a current of steam passed through the flask, and distilled as long as the distillate ceases to have an alkaline reaction. The distillate is rendered faintly acid with sulphuric acid, evaporated to dryness with sand, powdered hydrate of lime added, and the mixture extracted in Soxhlet's extraction apparatus with ether, most of the ether is distilled off

\* ANALYST, vol. xi, p. 16.

slowly, the remainder is allowed to evaporate spontaneously, and the residue is taken up with water and titrated with normal acid.

I have endeavoured in several ways to simplify the estimation, and especially to avoid the distillation. To my great regret, I did not succeed. I tried to shake the extract, dissolved in water and alkalised, with chloroform, ether, and benzol. The first two were utterly unsuited, because the mixture gelatinised so strongly that even after several days nothing had separated. Benzol forms a good deal less gelatin, but so many shakings were necessary that the labour was protracted unreasonably. (Kosutary states, on the contrary, that only three shakings are necessary.) I obtained, for instance, by shaking 8 grams. of extract, dissolved in 200 c.c. of water, and using 100 c.c. of benzol at a time, the following results :—

From the first shaking, the yield was 1.6 per cent. of nicotine; from the second, 0.9455; the third, 0.75; the fourth, 0.563; the fifth, 0.45; the sixth, 0.4277; with the probability of needing at least six more shakings.

Beckurts' recommendation to add alcohol to the chloroform, to prevent gelatinisation were followed, but with a negative result. This is due probably to the large amount of pectin present in the aqueous extract of tobacco leaves, while Beckurts' method works well with the official narcotic extract, which contains very little, if any, pectin.

After trying several modifications of the foregoing various methods, without arriving at any useful results, I finally decided upon the following, which is a simplification of Kissling's method, not being able, however, to do without distillation :—

Of an extract of tobacco leaves, 12.519 grams. was distilled in a current of live steam with hydrate of lime till the distillate ceased to give an alkaline reaction. The distillate, about 500 c.c., was faintly acidulated with diluted sulphuric acid, evaporated to 50 c.c., made alkaline with solution of soda, and shaken with ether in portions of 20 c.c. each time. The two liquids separated completely, and each three shakings were worked up together.

After evaporating the ether and the small amount of ammonia present, the residue was taken up with 20 c.c. of one-tenth normal sulphuric acid, any excess of the latter being re-titrated with one-tenth normal soda solution, using rosolic acid as an indicator. The first three shakings required 8.7 c.c. of soda solution. The remaining  $11\frac{1}{2}$  c.c. of one-tenth normal sulphuric acid correspond to 1.4635 per cent. nicotine. The next three shakings yielded 0.129 per cent., making altogether 1.5925 per cent. nicotine. Of the same extract 17.517 grams. were taken and distilled in a current of steam with solution of soda; about one litre of distillate was obtained, which was acidulated with sulphuric acid, evaporated, etc. The residue from four ethereal shakings was taken up with 10 c.c. of normal sulphuric acid, and re-titrated with normal soda, of which 8.35 c.c. was used. The remaining sulphuric acid corresponded to 1.526 per cent. nicotine. The next three shakings yielded 0.1186 per cent., making altogether 1.6446 per cent. nicotine. The difference between the two estimations is 0.0521 per cent.

Of another extract 17.859 grams. was distilled in a current of steam with soda solution, and gave one litre distillate. Treated similarly to the foregoing, the first three shakings required 7.974 c.c. of normal sulphuric acid, corresponding to 7.284 per cent. nicotine. The next three shakings yielded 0.19956 per cent., making altogether 7.484 per cent. nicotine.

According to my experience the use of Soxhlet's apparatus for the ethereal extraction, as recommended by Kissling, can very well be replaced with six shakings with 20 c.c. of ether each time.



## LAW NOTES. TO OUR READERS.

Some years ago it was decided to discontinue the reporting of ordinary police-court proceedings, and to give such cases as authoritatively established some point in connection with the working of the Acts which public analysts are interested. After a fair trial of this system, a majority of the members of the Society have expressed a wish that our old practice should be, to some extent, returned to, and, in accordance to such request, we have decided to resume the reporting of police proceedings. The line will be drawn at accounts of ordinary cases, and reports will only be inserted when any novel, legal, or chemical question arises, or where the certificate of the analyst is in any way attacked. Any member or subscriber desiring to be inserted with such a case is, therefore, invited to furnish us with a report of the proceedings, signed with his name, not for publication, but as a guarantee of exactitude.

**CHOCOLATE CHEWING-GUM.**—Wm. Burns, 42, William Street, Cowcaddens, and P. B. Jackson, 359, Queen Street, were brought before Sheriff Balfour at Glasgow on the 10th ult., charged with an offence under the Adulteration of Foods Act, by having, on the 2nd March last, sold a sweetmeat called chocolate chewing-gum, which was adulterated. Both parties pleaded guilty.

Mr. Wm. Kidd Smith, who appeared for the respondents, stated that the chocolate chewing-gum was sold as obtained from the manufacturers in Lincoln. The confection had its origin in America, and in this case it was mixed with a substance which could not properly be called gum, but had a rancid taste. The manufacturers were in Lincoln. The respondents stated that it was bought by children for the purpose of chewing and then making crackers.

At the request of the Sheriff, Dr. Russell explained that the substance complained of would not make crackers. There was nearly 42 per cent. of paraffin wax in it, and, as that matter was insoluble, would not combine with any of the fluids, and was a foreign body in the stomach. It might so irritate the stomach as to cause convulsions in the case of children of tender years.

The Sheriff said the article had a misleading name for the purpose of chewing and making crackers; and that children, seeing it labelled chocolate chewing-gum, might purchase it for eating. From Dr. Russell's description, it was perfectly clear that it was insoluble and indigestible, and would be hurtful to children. A trade like that must be put down, and accordingly he imposed a penalty in each case of £3.

The Sheriff afterwards explained to the respondents that unless they get a written guarantee from the manufacturers that the article was what it was represented to be they could be held liable, even though they sold it as obtained from the manufacturers.

Mr. Smith remarked that the agent who supplied the respondents had called in all the gum that he had supplied from the manufacturers in Lincoln.

**THE MARGARINE ACT.**—At Glasgow Sheriff Court, on the 11th ult., Sheriff Balfour presiding, James Easton and Charles Bell, provision merchants, 177, Parliamentary Road, were charged at the instance of Mr. Peter Pyfe, sanitary inspector, with having on Monday, 12th Feb., exposed margarine for sale by retail without having attached to each parcel, in such a manner as to be clearly visible to the purchaser, a label bearing in printed capital letters, not less than 1½ inches square, the word "Margarine," whereby, it being a first offence, they were liable, under the Margarine Act, 1887, to a penalty not exceeding £20. A plea of not guilty was tendered by the respondents, who were represented by Mr. James Dunbar, writer. Mr. G. Paterson, writer, appeared for the prosecution. Inspector Inglis stated that when he visited the shop he saw two boxes of margarine on the counter, and on the top of the boxes five lumps of margarine. On the top of these were seven lumps of butter. There was a ticket on the top of the butter, marked "Finest Irish." On a shelf behind the counter were 51 lumps of margarine without a ticket, and against them was a kit ticketed "Butter." He visited the shop in consequence of a complaint having been lodged at the Sanitary Office by a gentleman. In reply to Mr. Dunbar, witness said he held that there should be a ticket on each pile of lumps. There was a ticket lying on the counter in front of the margarine, as if it had fallen down. Inspector Armstrong corroborated, and in cross-examination expressed an opinion that in terms of the Act there should be a ticket attached to each lump. For the defence, William Wright, foreman to the respondents, and John Dawson, an employé of the firm, stated that on the day the inspectors visited the shop a ticket of the required size was attached to the margarine on the counter, and there were two tickets on the shelf. One of these was attached to the parcel that was being sold.

In giving judgment, the Sheriff said that when packages of margarine are exposed for sale they become parcels in the sense of the Act, and then they require to have larger labels attached to them. He would not hold that a shopkeeper is bound to attach to a small piece of butter a large label. If a lot of little lumps are put into one heap, and on it is distinctly put a label marked "Margarine," he would hold that to be a sufficient compliance with the Act. Retailers ought to see that each parcel is marked with a distinct label. The respondents had clearly violated the Act, but it might be that they thought they were carrying out its provisions. He would give them the benefit of the doubt, but he hoped that they, and shopkeepers in general, would know better in future. The Sheriff inflicted a penalty of 10s., which was paid.

**THE CORK COFFEE CASE.**—In this case, referred to in our last issue, the Recorder has altered the decision on appeal, and acquitted the defendant, thus ignoring the English decision of *Liddiard v. Rees*.



## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

## INSTITUTE OF CHEMISTRY.

To the Editor of the ANALYST.

SIR,—As the editor of the *Chemical News* feels that it is necessary, from certain reasons, to close the correspondence in reference to the Institute of Chemistry, I shall feel obliged if you will allow me space to reply to two letters which appeared in a recent number of that journal. The following is, in effect, a copy of the letter which I sent:—

"Having been away from home I have only now been able to read the replies (*Chemical News*, vol. lvii., p. 120) to my letter in the previous issue. In one of them I am struck with the fact that there are persons who see meanings in a sentence which the language does not imply, and which the writer of the sentence did not intend. Such persons usually support their arguments by extracts from the poets, and by an artistic admixture of Latin or dog-Latin quotations. I do not say that Mr. Friswell, the writer of one of the letters, who employs poetical Latin quotations, is one of those gentlemen, but I do say that he has misread my letter when he assumes that in giving the number of professors, and ex-professors, non-professors, etc., on the council, I desired that they should be removed from it. My statements and figures simply showed that the number of professors is excessive as compared with those representing the other branches of the profession.

"A gentleman at the annual general meeting said he was told that a number of the voting-papers were counted as having opposed the Council list, in which my name alone was removed. I asked one of the gentlemen who had been engaged in opening the voting-papers if that were true, and he replied that it was not, and that all the papers he saw had a number of names scored out, and a corresponding number inserted in lieu thereof, and these names were all, or nearly all, those suggested in the circular letter bearing my name. I think I am therefore justified in believing that another of the *Chemical News* correspondents, 'One who was there' (although he does not say where) was misled respecting the number of proxies in favour of the president and secretary on the one hand, and of my friends and myself on the other.

"Mr. Friswell and the correspondent 'who was there' consider that the course advocated by my friends and by me will not be likely to maintain the dignity of the Institute, whilst we, on the other hand, believe that our ideas, carried out, would place the Institute in a much more dignified position than it is under the present conditions.

"I think the criterion of real knowledge and ability is higher than that of having attended special courses of lectures. A gentleman told me he wished, a few weeks ago, to engage a senior assistant, and interviewed one who was already either a Fellow or an Associate of the Institute, and who had had a regular training in one of the specified colleges (which would have been regarded by Mr. Friswell and his friends as excellent). He asked him what was the action of hydrochloric acid on ferrous sulphide, and the 'regularly' trained student replied that it would form sulphuric acid. Other questions, equally simple, were answered with a somewhat similar approach to accuracy.

"We do not say that a student should not attend lectures at any of the specified colleges—quite the contrary—all we say is that, provided he can *show* that he has gone through a regular course of study in theoretical and practical chemistry, under an efficient teacher, who is not a mere crammer, he should be admitted to the examination of the Institute, which should be sufficiently searching to make the membership of the Institute a criterion of education and ability, and not a mere guarantee that the members have attended one or more of twenty or thirty specified schools or colleges.

"If those who have such high opinions of what can be achieved in universities and colleges by means of lectures, and for which kind of training they desire to capture and apply the word 'regular,' will speak to the average medical man on any subject in which he has been 'regularly' trained, he will probably arrive at the conclusion that the training had been exceedingly superficial.

"Attendance at lectures at the universities and colleges is, no doubt, the most convenient way of acquiring knowledge of theoretical chemistry, but it is not necessarily the most efficient, and at the present time, as it is not compulsory for an analyst in practice to be a member of the Institute, I think it would be more dignified if the Institute demanded simply (at all events for a few years, as a trial) that the student should be admitted to its examinations if he can give evidence of his having been efficiently and regularly trained in theoretical and practical chemistry, physics, and mathematics, and of his having received a satisfactory general education, and that the question of his having attended one of a specified number of colleges be not held of primary importance.—I am, etc.,

Royal Institution Laboratory, Manchester.  
13th April, 1888.

"WILLIAM THOMSON"

## APPOINTMENTS.

Dr. William R. Smith has been appointed public analyst for the city of St. Albans; Dr. William Johnstone has been appointed public analyst for Dunstable.



# THE ANALYST.

JUNE, 1888.

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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A GENERAL meeting of the Society was held at Burlington House, on the 9th ult., the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election:—

As members—Mr. W. F. Lowe, F.C.S., public analyst for Chester; Dr. F. L. Sed, F.I.C., public analyst for Islington; Mr. J. Bemrose, Montreal. As associates—Mr. W. D. Mildred, assistant to Mr. A. C. Wilson, Middlesbrough; Mr. F. W. Richardson, assistant to Mr. Rimmington, Bradford.

The following papers were read and discussed:—

"Some Effects of Food Preservatives on the action of Diastase." By Dr. H. Leffmann and W. Beam, in connection with which the President communicated a "Note on the Detection of Saccharine in Beer."

"Note on the Wear and Tear of Platinum Dishes." By Dr. Vieth, F.C.S., F.I.C.

"Notes on Liquorice or Spanish Juice." By Bernard Dyer, B.Sc., F.C.S., etc.

Mr. B. Blount exhibited a new form of fat extraction apparatus and explained its working.

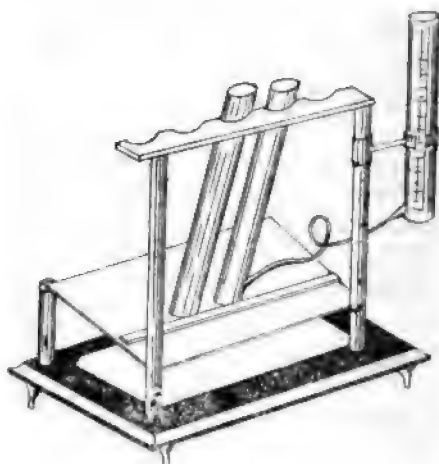
The next meeting of the Society will be held at Burlington House on Wednesday, the 13th inst., at 8 o'clock.

## AN APPARATUS FOR COMPARISON OF COLOUR TINTS.

By A. W. STOKES, F.C.S., F.I.C.

THIS apparatus (which was shown and explained at the February meeting) was especially devised for Nesslerising in water analysis, but is of use in such operations as determination of lead in water, carbon in steel, colour of sugar, etc.

Whenever it is necessary to compare a number of depths of tint with one another, it is very inconvenient to have to make up a large series of standard tints. The apparatus obviates this necessity. By reference to the annexed diagram\* it will be seen to consist of a base-board, on which lies a sheet of white opal glass. Placed obliquely a little way above this is a sheet of colourless glass, on which rest the Nessler-tubes containing



the solutions whose tints are to be compared. The mouth ends of these tubes recline towards the observer, in grooves hollowed in a horizontal bar. This bar is supported by two pillars, on one of which slides a ring claspings a calibrated glass tube; this tube has a side tubulure at the base, and is thus by means of a short india-rubber tube connected with a similar but uncalibrated tube resting on the glass plate. A known quantity of the standard colour being placed in that tube carried by the ring and diluted up to the top mark with water, if this tube be raised in its sliding collar its standard solution will flow into the tube resting on the glass plate. The movable tube is so calibrated as to show how much liquid is present in the tube connected with it. Hence, if a Nessler-tube containing liquid of an unknown depth of tint be placed beside the tube resting on the glass plate, and the tube in the sliding ring be raised or lowered till the tints agree in the two tubes that rest on the glass plate, a simple inspection of the level of the liquid left in the calibrated tube will give the quantity of standard colour the unknown solution is equal to. The distance apart of the grooves in the horizontal bar is the average width between the eyes, so that as the mouths of the tubes lie inclined towards the observer, and the light is reflected up from the opal glass through the full length of the tubes, observations are easily made. As shown the apparatus will take three tubes for comparison with the standard colour, but it may readily be made to take any

\* By kindness of the Editor of the *English Mechanic*.



number. By putting a similar pair of connected tubes on the other pillar an unknown tint might be placed between *two* known depths of tint. A small slip of wood is fixed along the glass plate to keep the tubes in an inclined position. By placing blackened cards in front and behind the tubes light can be prevented from entering the tubes except from the bottom. The apparatus is simple and readily cleaned. It can be obtained from Messrs. Townson and Merse.

#### EFFECTS OF FOOD-PRESERVATIVES ON THE ACTION OF DIASTASE.

By HENRY LEFFMANN AND WILLIAM BEAM.

(Read at Meeting, May, 1888.)

THE use of antiseptics in perishable articles of food has become quite general in recent years, and has been, to a certain extent, the subject of legislation. Salicylic acid has been, probably, the most used, and while sanitary authorities of different countries have, as a rule, opposed its use, there has been no positive evidence of its injurious action, even when continued for some time. Lehmann published in Pettenkofer's "Archives of Hygiene" several instances in which healthy male adults had taken for many days considerable doses of this acid without apparent injury. While there may be a legitimate field for the use of the agents in articles of food of a highly perishable character, and especially where the addition is made known, there can be no question that their indiscriminate use is dangerous. Independently of any directly injurious action, it is important to inquire how far they may interfere with the nutritive or medicinal value of any articles with which they may be associated. The matter has been brought prominently to our notice in consequence of some analyses made by us for the State Board of Health of Pennsylvania, in which the free use of salicylic acid in beers and malt extracts was detected. Similar results in regard to beers were found by other State Boards and the Department of Agriculture of the United States Government. It becomes important, then, to inquire how far the presence of these substances may interfere with the diastasic action ascribed to preparations of malt. It must be noted that with a number of the malt extracts now on the market the addition of a preservative has very little significance, because, as prepared, these articles are merely weak beers, and possess no diastasic power. Thus, of eleven samples tested, including all the extracts widely known in this market, only four had any appreciable effect, and but one of these was strikingly efficient.

We have undertaken a few experiments to determine what retarding effect the common food-preservatives may possess. The method of operating was that indicated by Drs. Duggan and Coale in papers in the *American Chemical Journal* and elsewhere, based on the estimation of the sugar formed in presence of a large excess of starch. Arrow-root starch was selected for reasons given by these observers. To avoid error due to varying action of the dilute solutions of malt which are required for the experiments, a blank experiment was made in each set, and from this the diastasic value of the pure malt extract was determined. In the first observations maltine was employed, it having been shown by our previous tests to be by far the most active of the commercial extracts, but later Schuchardt's diastase was used. The Fehling's solution was prepared as directed by Allen, and the determinations were made volumetrically. Many of the experiments

were duplicated, with concordant results. In addition, a number of tests were made by the method given by Allen for valuing malt-extracts. The results obtained were similar to those given below, but, not being capable of quantitative comparison, are not detailed here.

The antiseptics selected were salicylic acid, boric acid, sodium acid sulphite, saccharin, beta-naphthol, and alcohol. The sample of beta-naphthol was of the form now sold under the name hydronaphthol. In all the experiments the temperature, time of action, strength of starch solution (30 grammes to litre) were the same.

In the following table the effect is represented produced by 0.5 c.c. of maltine diluted to 5 c.c., and added to 100 c.c. of starch solution. The figures give the proportion of antiseptic used to the whole volume of solution, and the amount of sugar formed from the starch—that contained in the maltine being deducted:—

Antiseptic used.	Proportion.	Fehling's Solution required.
None		245 c.c.
Salicylic acid	1 to 500	No sugar formed.
" "	1 " 1,000	" " "
" "	1 " 20,000	245 c.c.
Boric acid	1 " 1,000	245 "
Sodium acid sulphite	1 " 1,000	245 "
Saccharin	1 " 1,000	18.5 "
"	1 " 500	5.6 "
Beta-naphthol	1 " 1,000	204 "
" "	1 " 500	174 "
Alcohol	1 " 25	245 "

In the above experiments the antiseptics were added to the starch and the maltine allowed to act at once. In the following, very small quantities of salicylic acid and alcohol were first mixed with maltine and allowed to stand four days before addition to the starch.

Antiseptic used.	Proportion.	Fehling's Solution required.
None		245 c.c.
Salicylic acid	1 to 20,000	174 "
Alcohol	1 " 500	221 "
Salicylic acid	1 " 20,000 }	174 "
Alcohol	1 " 500 }	

As before, the proportion of antiseptic is given to the whole volume of solution, after addition of the starch.

#### EXPERIMENTS WITH DIASTASE.

Proportion of Diastase.	Antiseptic.	Proportion of Antiseptic.	Fehling's Solution required.
1 to 500	None		300.5 c.c.
1 " 500	Salicylic acid	1 to 3,000	286 "
1 " 500	" "	1 " 1,500	16 "
1 " 500	" "	1 " 1,000	No sugar
1 " 1,000	None		263 c.c.
1 " 1,000	Salicylic acid	1 " 1,000	No sugar
1 " 2,000	None		238 c.c.
1 " 2,000	Salicylic acid	1 " 5,000	82 "
1 " 2,000	" "	1 " 3,000	No sugar
1 " 1,000	Boric acid	1 " 1,000	250 c.c.
1 " 1,000	Sodium acid sulphite	1 " 1,000	263 "
1 " 500	Saccharin	1 " 1,000	86.3 "
1 " 1,000	" "	1 " 1,000	No sugar
1 " 1,000	Betanaphthol	1 " 1,000	238 c.c.
1 " 1,000	Alcohol	1 " 25	250 "



Saccharin was included in the above experiments on account of statements made recently in medical journals that it has decidedly antiseptic powers, and that it may be used liberally for internal administration. Such an opinion would be likely soon to lead to its use as far as its taste would permit.

The inferences from the above observations are that salicylic acid is especially objectionable in a malt extract, and that saccharin is also unsuitable. Alcohol in the extract is also objectionable. Betanaphthol has some retarding action, but not very considerable. Boric acid and sodium acid sulphite seem to have but little retarding effect. Their effect, however, by long-continued action on the malt extract before adding it to the starch has not been tested. It is obvious that these conclusions apply to the use of preservatives in prepared articles of food.

With a view of adding further to this point, a sample of Fairchild's pancreatic extract was examined as to its action on starch, with and without addition of salicylic acid. 0.3 grm. pancreatic extract in 100 c.c. starch solution formed sugar equal to 161.3 Fehling's solution; in presence of one-thousandth part of salicylic acid, no sugar was formed.

The President said that the paper just read was a very important one in many respects, and he was glad to think that members of the Society so far distant as Philadelphia had made such a valuable contribution to the proceedings.

#### THE DETECTION OF SACCHARINE IN BEER.

By A. H. ALLEN, F.C.S., F.I.C.

(Communicated to Meeting, May, 1888.)

CLAUSE 5 of the Customs and Inland Revenue Act has special reference to the use of saccharine as a sweetener and preservative of beer, and, in the discussion of the Bill in Committee, on April 27th, Sir Lyon Playfair, who was supported by Sir Henry Roscoe, contended that it was a mistake to prohibit the use of saccharine instead of sugar, as such a course would have serious deterrent results on a growing industry. He stated that foreign brewers were using saccharine largely, and that it could be manufactured in this country at a cost of 20s. to 30s. per pound. He concluded by asserting that the presence of saccharine in beer was very difficult to detect, and that there was another substance which, if employed in conjunction with saccharine, would absolutely prevent its detection, and therefore the result would be that if its use were not regulated the Excise would be defrauded. In consequence of the last statement I wrote to Sir Lyon Playfair asking what was the substance which would prevent the detection of saccharine if used in conjunction with it, and learned that the remark had applied to salicylic acid. Under these circumstances it is worth while to review shortly the analytical characters of saccharine.

Falberg's saccharine, we all know, is a coal-tar product, of which the systematic name is benzoyl-sulphonic-imide. It is the anhydride of orthosulphamidobenzoic acid, and forms a series of salts as sweet as, if not sweeter than, itself. The sweet taste of saccharine is variously stated at from 130 to 330 times that of cane sugar. As a matter of fact,

some saccharine tabloids, each of which was said to be equivalent in sweetening power to a lump of sugar, were found to have an average weight of 0.45 grain each, while an average lump of sugar weighed about 90 grains. This relationship indicates that the saccharine has about 200 times the sweetening power of sugar.

When heated, saccharine partially sublimes, giving vapours of an intensely sweet taste. If heated in admixture with caustic soda to 250° C. it forms salicylic acid, which can be detected by dissolving the residue in acidulated water, agitating with ether, evaporating the ethereal solution to dryness, and adding ferric chloride, when the characteristic violet coloration is produced. When saccharine is ignited in admixture with caustic or carbonated alkali, preferably with addition of a little oxidising agent, such as nitre, it yields a residue containing sulphate, which of course can be detected by adding chloride of barium to the acidulated solution. The weight of sulphate of barium precipitated, if multiplied by 0.785, gives the weight of saccharine to which it corresponds.

An aqueous solution of saccharine when heated with potassium ferricyanide becomes coloured apple-green, an odour of hydrocyanic acid being evolved.

Saccharine is extracted with tolerable facility on agitating its acidulated aqueous solution with ether.

Utilising these reactions I have found no difficulty in detecting less than  $\frac{1}{2}$  grain of saccharine in one pint of beer. The beer was concentrated to about one-third, and at once agitated with ether. As a matter of fact, I added a little phosphoric acid, but this precaution was superfluous, as the liquid had already a marked acid reaction. Acidulation with sulphuric acid is not desirable. The ethereal solution left on evaporation a residue which was, of course, intensely bitter and was not adapted for the recognition of saccharine by any test other than the production of sulphate on ignition in the presence of alkali. By this method, however, distinct evidence of the presence of saccharine was obtained, whereas a blank experiment on the same beer, to which no saccharine had been added, failed to show any trace of sulphates by the same process.

A purer residue is obtained by treating the beer with acetate of lead and filtering before agitating with ether. There is no occasion to remove the excess of lead, and in fact the use of sulphuretted hydrogen would be very undesirable.

It will be seen that the detection, and even the determination of saccharine in beer presents no great or insurmountable difficulty, even in the presence of salicylic acid. I do not know of any organic substance containing sulphur, which would be likely to be used in or be present in beer, which would be extracted by ether from its acidulated solutions, and hence be likely to interfere with the above method of detecting saccharine. The Inland Revenue chemists have expressed the opinion that they would be able to recognise saccharine in beer by its taste. This might be so in the absence of hop-substitutes, but the presence of quassia would produce an ethereal extract of so intensely bitter a taste as wholly to mask the sweetness due to saccharine. At any rate, if the Inland Revenue chemists have devised any process by which the saccharine can be isolated from beer in all cases in such a state of purity as to be recognisable by the taste it is to be hoped that they will depart from their usual practice and communicate their knowledge to their brother chemists.

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## DISCUSSION ON MR. RIMMINGTON'S PEPPER ADULTERATION AND PEPPER ANALYSIS PAPER.

*(This paper appeared, with illustrations, in our May issue.)*

Dr. VIETH asked the author of the paper whether he was only able to ascertain the presence of the adulterant, or to make a quantitative statement; he (the speaker) thought this was very desirable; in fact, analysts were obliged to state the quantities in their certificates.

Dr. DUPRE said that the Act did not really require in all cases the amount of adulteration to be stated; that was in a great measure for the magistrates, who wanted to have some idea so as to apportion the fine in accordance with the extent of the adulteration. The Act really requires us only to state whether or no the article is of the nature, substance, and quality demanded, but magistrates nearly always required quantities to be stated.

The same principle obtained as regarded poison; it did not matter whether one found one hundredth of a grain or a pound; the question was the presence or absence of the poison. At the same time the question was of considerable interest, and he would like to know how Mr. Rimmington would word his certificate in the case of pepper.

Mr. HEHNER said that, apart from the detection of long pepper, the most important question was, whether it should be held to be adulterated, if sold as pepper, without notice to the purchaser. He thought that legitimate trade would be most seriously interfered with if it were so held. Unless a purchaser asked for round pepper, he saw no reason why he should not be supplied with long pepper, both being pepper, precisely as a vendor might supply any kind of jam to the purchaser asking for jam only, not particularly stating the kind required. The same remark applied to very many articles of consumption: the purchaser should particularly state his requirements. It is no adulteration to sell white mustard or black mustard, although both differed essentially, provided always that mustard only, not any specified kind, is asked for.

It seemed to him very unwise to interfere with legitimate trade; the aim of analysts should be to educate the public; the trade would then in time be obliged to adopt the practice which he thought was an ultimate necessity, namely, that each article should be labelled with its composition. Certain firms in the coffee trade had already adopted it, stating distinctly: "This article contains so much coffee, and so much chicory." The same principle should be extended to other articles, and not any difficulties laid in the way of sale.

Mr. DYER said that the questions of chicory and coffee, and pepper were quite different. The chicory mixture was made by the grocer. The pepper mixture of pepper and long pepper was made by the wholesale man. The honest wholesale houses were greatly aggrieved at the dishonest houses doing this, and so underselling them by a small sum per lb., and the honest houses quite opposed the idea that long pepper was legitimately called pepper at all. There had been prosecutions for selling long pepper, and convictions were obtained, and a large firm had told him that the price of long pepper had since in consequence dropped enormously, showing that there was no legitimate demand for it. It seemed to him that it was like the case of the deliberate incorporation of a small quantity of water in lard. No one contended that the pur-

chaser was much prejudiced by that, but it made a difference in the wholesale price of the lard, and enabled one man to undersell another.

Dr. DUPRE was rather reluctant to differ from Mr. Hehner, but he should like to protest against his remarks as to custom of the trade. There was no abomination that could not be covered by what was called the custom of the trade, and therefore an analyst has no business to be governed by it so long as he believes that the sample is not of the nature, substance, and quality demanded.

That, of course, is different to the question as to whether long pepper is an adulteration—long pepper would not be sold as pepper by itself, and therefore, if they got it mixed with the genuine article he was inclined to look upon it as an adulteration.

With regard to another remark made as to the public requiring these mixtures—the poor public are made to swallow many things on the plea that they like it—they don't like to have pure coffee, pure violet powder, for instance. He maintained that a public analyst had no right to take the taste of the public into consideration when he has to decide the question of adulteration. If the public require an adulterated article, let it be sold as adulterated, but the public analyst had nothing to do with that.

Mr. CASSAL agreed very strongly with Dr. Dupre. The question of public taste was one that ought not to govern them at all. It was part of their business to teach the public what was good for them, and they did so by certificates and prosecutions as far as they could. He thought there could be no question that long pepper was an adulteration; it contained, according to most analyses, about 2 per cent. of piperin, which was a valuable constituent of pepper, which contained at least 5 to 6 per cent., and an admixture of long pepper must be regarded by all sensible public analysts as an adulteration. The question as to deciding the quantity of an adulterant for insertion in a certificate was a most important one, although technically the schedule to the Act did not require that the quantities should be stated absolutely.

In view of the present state of the public knowledge and the gross ignorance of those who administered the law, it was most desirable that they should be guided, as far as possible, by the public analyst's certificates, and, although it was not very scientific, he was in favour of using the term "*at least*" so much, in regard to adulterants detected. He thought it was desirable in the case of adulteration of pepper with long pepper to state a minimum percentage of adulteration on the results of both microscopic and chemical examination. The difficulty was very great, it being impossible to tell the exact percentage of adulteration, either with rice or long pepper.

Mr. RIMMINGTON quite agreed with Mr. Hehner about being over virtuous, respecting certain articles, such as coffee. He ventured to say that 99 out of 100 people who asked for coffee and had genuine coffee supplied them, would take it back or at any rate not go to the shop again. Of course there were people who would have genuine coffee, but that was quite different from the general run of society.

Then again with regard to mustard; no doubt a better mixture could be made with flour and black mustard, than with white mustard only. There is, however, a limit to all things, and if cayenne pepper be used that would constitute an adulteration.

Another article was ground ginger, which is mixed with exhausted ginger, and it is *not possible for any one to tell what amount of exhausted ginger has been introduced.*



This refers more particularly to the commoner kinds of ground ginger and not to the better qualities.

In answer to some observations as to the estimation of the amount of adulteration in pepper, Mr. Rimmington said that all one could do was to make as good a guess as one could. In a certificate he would say "I am of an opinion it contains about so much of so and so."

Another question arose whether this adulteration of pepper was to be regarded as an offence against the public. Of course, commercially it is a fraud, but in an ounce of pepper sold to any one the amount of injury is very small. The article sold answers its purpose; the trade itself is the chief sufferer from illegitimate competition.

Mr. STOKES remarked that it was useless to grind up pepper mixtures very fine in order to use high microscopical powers so as to distinguish between ordinary pepper, long pepper, and rice, because by so doing the ultimate starch granules alone can be compared; and in a mixture containing all three of these the ultimate starch granules of the long pepper vary from the size of those of the ordinary pepper to those of rice. Mr. Stokes said that he had noticed no starch granules that either in shape or size were peculiar to long pepper.

A method that at once shows any admixture of long pepper or rice is to take the pepper as ground up for sale, place a small portion of it in a drop of glycerine on a slip of glass and under a cover-glass. Place this beneath the microscope, having a one-inch power, and provided with a polariscope so arranged that the Nicol prisms are crossed, giving a dark field. Now, if the slide contain only white or black pepper the field will remain dark; if there be any rice or long pepper these will alone be visible, giving a sort of ghostly white image. Nothing else gives the same or a similar appearance. The number of such particles may be counted and their proportion so estimated. There is a difference in the image given by rice and that given by long pepper, but it requires a practised eye to distinguish. If, however, another portion be taken and be viewed dry, as an opaque object, under the one-inch power, should there be any rice present the larger particles of this will show as structureless, opalescent particles, looking very like pieces of broken paraffin-wax.

If ground up till only the ultimate starch granules are visible none of these effects will be seen.

For the detection of rice and long pepper a higher power than the one-inch is neither necessary nor advisable.

As to whether long pepper could be called an adulteration, Mr. Stokes very much doubted, for Mr. Rimmington, in his paper, stated that there were eight different kinds. So long as pepper was not produced by one plant only, and that the berry contained the essential constituents of pepper, that is, the peculiar resin and piperin, it seemed impossible to him to say that such a berry was not "pepper."

Mr. RICHARDSON (who had read the paper for the author) said that he submitted that Mr. Stokes's process for the detection of starches was not scientific; it depended on certain refractions of light, and he contended that it was impossible to come to any definite conclusion respecting such minute bodies as starch granules in pepper, and in a majority of cases no one would like to certify that a sample was adulterated upon such evidence only.

He must say that in regard to detecting rice with the polariscope, that it was

absolutely impossible. He submitted that if you took a small quantity of pepper, and examined it in the ordinary way, under the microscope you would discover very little indeed, but if washed in the manner that has been described, there would be obtained three strata in the deposit, each of which could be separately examined. All they could do by Mr. Stokes's method was to judge by certain refractions of light and that was all. He hoped, however, they would all try it.

Mr. RIMMINGTON's object in bringing the matter before the Society was to ask the members if the cells described and exhibited were not those of long pepper. If so, what were they? With regard to the difference between white pepper and long pepper, any one taking pepper as a condiment would perceive the remarkable difference at once, and no one would like to use the latter with meat. In answer to Dr. Vieth

Mr. RIMMINGTON said he was rather of opinion it was not possible to say what the proportion of adulteration might be; it was enough to say that it was adulterated; of course in the eye of the law it is a matter of indifference whether the quantity was 5, 10, or 15 per cent., if you can say with certainty that it is sophisticated. He did not think it possible to say to what extent a sample might be adulterated. You might think you could estimate the starch, but could you be certain that all pepper contains the same amount of starch—might as well say that all grapes were equally sweet, or all apples equally sour. Then, again, with regard to piperin, there were similar wide variations in the proportion in pure pepper. Therefore, as far as chemical analysis was concerned it was impossible to come to any more definite conclusion than by the microscope, although certain matters may be determined by analysis. He had shown that if the sample were shaken up with alcohol and water, as described, the adulterant would settle in a distinct layer, and an approximate judgment may be formed. The amount of adulteration that had hitherto existed was not large, but for the last twelve months he had scarcely met with a sample of pepper that had not more or less of what he believed to be long pepper.

Mr. HEHNER said that his expression of opinion was a very different one from that attributed to him by Mr. Rimmington; he objected most strongly to the sale of chicory as coffee; every article should be sold on its own merits, and not under false colours.

Dr. VIETH, in conclusion, said the hearty thanks of the meeting were due to Mr. Rimmington for his very interesting paper.

*(Conclusion of the Society's Proceedings.)*

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#### DETERMINING THE FIXED ACIDS IN BUTTERS AND MARGARINES.

By THOS. P. BLUNT, M.A., F.C.S., F.I.C.

It is obviously an advantage in examining "margarines" to determine the insoluble acids; this is especially the case with mixtures containing a proportion of genuine butter; for, corroborative proof is here of the greatest importance.

I am in the habit of utilising the residue from Reichert's process for this purpose, and have found the method work so well and accurately that I venture to bring the details before the readers of the ANALYST. I use a very slight excess of sulphuric acid in destroying the soap previously to distilling, to obviate charring when the fluid in the flask becomes concentrated, and I add two or three pieces of pumice, according to the usual practice, to prevent bumping. When the distillation is complete I filter the distillate (which is nearly clear with pure butter, but full of white crystals where "margarine" is under examination) and reserve the filter; the flask (about 300 c.c. capacity)



now nearly filled with distilled water, boiled, and cooled; this is repeated, and the water poured from the cake of fat: the flask is now reversed on a ring of the retort-stand, over the reserved filter, and allowed to drain. The flask and its contents are then heated in the water oven, the clear, fatty acids poured from the pumice into a flat glass of the usual form, the pumice and flask washed two or three times with a warm mixture of equal parts of ether and alcohol, which is passed through the filter into the glass containing the fat. The solvent is then evaporated off, and the acids dried and weighed as usual.

There is little new in the above, but it is a decided saving of labour over a second saponification, and the results are exact; a genuine butter gave a distillate requiring 6 c.c. of decinormal soda for saturation, and fixed acids 87.9 per cent. One obvious advantage of the modification is the removal of the greater part of the volatile acids by distillation, and the comparatively slight washing of the fixed acids required.

#### VOLUMETRIC ESTIMATION OF ZINC.\*

R. BENEDIKT AND M. CANTOR.

CONTRARY to Classen's statement that zinc oxide cannot be properly titrated with normal acid and alkali, we have found that the oxide and the carbonate can be accurately titrated with methyl-orange as indicator, and soluble zinc salts by means of phenol-phthalein. For our experiments we used the materials which we happened to have in stock, viz.:—

1. Hydrochloric acid: 1 c.c. =  $\cdot 0333 \text{ Na}_2\text{CO}_3 = \cdot 02544 \text{ ZnO}$ .

2. Sodie hydrate; checked with the said acid, with the following results:—

Using methyl-orange: 1 c.c. acid = 1.432 c.c. soda.

Using phenol-phthalein: 1 c.c. acid = 1.462 c.c. soda.

3. The double salt of zinc and potassic sulphates:  $\text{K}_2\text{SO}_4, \text{ZnSO}_4, 6\text{H}_2\text{O}$ , which we purified by recrystallisation, and then analysed by the usual methods to ascertain its purity. The zinc oxide got by this analysis, .210 grm., was dissolved in 20 c.c. of the acid, and after adding methyl-orange titrated back with 17.2 c.c. soda, which was .2086 grm. ZnO or 18.17 per cent. instead of 18.27 (in the double salt).

We have made a series of experiments in this way. A weighed quantity of the double salt was dissolved in a porcelain dish in boiling water, and after adding a few drops of phenol-phthalein, sodie carbonate was added until the liquid turned reddish. The precipitated zinc carbonate was then collected on a filter, washed with boiling water, then redissolved and titrated as before.

Weight of double salt.	Hydrochloric acid. 1 c.c. = $\cdot 02544 \text{ ZnO}$ .	Zinc Oxide.	
		Per cent. found.	Per cent. calculated.
2.042 grms.	14.55	18.12	18.27
3.247 „	23.10	18.10	18.27

\* *Zeitschr. f. angew. Chemie*, No. 8.

The volumetric estimation of zinc in combination with the stronger acids is easily performed in the following manner:—

The strongly diluted solution (about  $\cdot 1$  ZnO in 50 c.c. water) is mixed with phenolphthalein, and titrated with standard soda, until the liquid turns intensely red. A few more c.c. of soda are now added, the liquid boiled for some time, and then titrated back with standard hydrochloric acid. The following experiments show the accuracy of the process:—

Weight of double salt.	Soda solution. 1 c.c. = $\cdot 0174$ ZnO.	Hydrochloric acid. 1 c.c. = $1\cdot 462$ c.c. soda.	Zinc oxide.	
			Per cent. found.	Per cent. calculated.
3.0053	36.2	3.07	18.36	18.27
3.3830	80.3	30.40	18.47	18.27

In applying the process to zinc ores, the zinc must be, as usual, freed from other metals, and precipitated as carbonate, which is then dissolved in standard acid, etc. The volumetric process gives results far more accurate than the gravimetric one, as there are less sources of error. It is well known how difficult it is to get rid of the last traces of alkaline salts, the burning of the filter with ammonic nitrate is troublesome, and the zinc oxide is often contaminated with silica.

In analysing zinc salts with methyl-orange as indicator, both the free acid and oxide may be readily estimated, and the total amount of zinc oxide may then be rapidly estimated by the use of phenolphthalein. We now give an example. Analysis of commercial zinc chloride.  $1\cdot 463$  grms. of the salt was treated with hot water, which left a small quantity of insoluble residue. After adding methyl-orange, it took 1.3 c.c. of our hydrochloric acid, which equals 2.26 per cent. of free zinc oxide. The liquid, which was now quite clear was, after addition of phenolphthalein, mixed with 50 c.c. of the soda, then boiled and titrated back with 1.2 c.c. of acid, which makes the total zinc  $46\cdot 04$  per cent. For further control we added hydrochloric acid until the precipitate was quite dissolved, heated to boiling in a basin, and added sodic carbonate until the liquid turned red. The precipitated zinc carbonate, after being thoroughly washed, took  $32\cdot 95$  c.c. acid =  $45\cdot 97$  per cent. zinc. To make still more sure, the chlorine was also estimated. Found  $48\cdot 37$  per cent. Calculated from the combined zinc,  $48\cdot 28$  per cent. Our process is more particularly valuable for the estimation of the free zinc oxide in commercial samples of zinc chloride, its gravimetric estimation being of course much more troublesome and liable to inaccuracy.

#### ON CERTAIN SOURCES OF LOSS IN THE DETERMINATION OF NITROGEN BY SODA-LIME.

\* BY W. O. ATWATER AND E. M. BALL.

THE object of the following experiments was to test the effect of open space between the soda-lime and the upper wall of the tube (channel, as often recommended), and of lengthened time of sojourn of gases in the combustion tube, upon the amount of

\* *American Chemical Journal.*



nitrogen obtained in the form of ammonia in the determination by the soda-lime method. The determinations were conducted by the method described in a previous article as usual in this laboratory, except that:—

1st. In some cases the tube was packed so full with soda-lime as to leave no considerable channel, in accordance with our usual practice, while in others less soda-lime was used, enough to fill perhaps from four-fifths to two-thirds of the whole interior of the tube. The channel left was thus quite large.

2nd. The anterior layer consisted in some cases of finely powdered soda-lime like that with which the nitrogenous substance is mixed for combustion, instead of the coarse particles with which (to secure more contact between the passing gases and the soda-lime, and hence more complete ammonification) we commonly fill the anterior part of the tube.

3rd. In some cases the combustion tube and the anterior layer of soda-lime were made longer than usual.

4th. The combustion in some cases occupied the usual time, about three quarters of an hour; in others it was prolonged to two and a half hours, as is done by some chemists.

It seemed desirable that the trials be made with a substance which had been found by other observers frequently to fail to yield all its nitrogen in the form of ammonia. For this casein was selected.

To test the accuracy of the nitrogen determinations when made by soda-lime in the usual way, comparative tests were made with Kjeldahl's method in two specimens. In the determinations with soda-lime, the precautions as to compact filling of tube, heating of anterior layer before applying heat to mixture of soda-lime and nitrogenous material, moderate heating throughout, and moderate time of combustion, previously mentioned as usual in this laboratory, were observed. The results of these comparative tests are stated in Table I.

TABLE I.—*Nitrogen obtained from Casein by Soda-lime and Kjeldahl Methods.*

Casein.	Nitrogen found, per cent.			
	Soda-lime Method.		Kjeldahl Method.	
	Separate Determinations.	Average.	Separate Determinations.	Average.
First specimen ..	12.20 12.10 12.10 12.05	12.11	12.07	12.07
Second specimen ..	12.44 12.43		12.07 12.44 12.41 12.43	

The determinations in the "first specimen" were preliminary, and the duplicates with soda-lime are somewhat wide apart. The "second specimen" was used for the determinations of Table II., in which those of series A are the same as given in Table I.

TABLE II.—*Nitrogen Determinations in Casein by Soda-lime under Different Conditions.*

Series.	Length of Tube.	Anterior layer of Soda-lime.		Packing of Tube.	Time of combustion, approximate.	Colour of acid after combustion.	Nitrogen found.
		Length.	Kind.				
A	cm.	cm.					per ct.
	38	12	Coarse.	No channel.	45 minutes.	Nearly Colourless.	12.44
	38	12	"	"	"	"	12.43
B	38	12	Fine.	Channel.	"	"	12.25
	38	12	"	"	"	Slightly coloured.	11.85
	38	12	"	"	"	Very dark.	11.66
C	38	7.5	"	"	"	Amber coloured.	12.38
	38	7.5	"	"	"	Colourless.	12.00
	38	7.5	"	"	"	Very dark.	10.51
D	38	12	Coarse.	No channel.	2½ hours.	Slightly coloured.	12.27
	38	12	"	"	"	"	12.05
	38	12	"	"	"	"	12.05
	38	12	"	"	"	"	11.96
E	38	12	Fine.	Channel.	"	"	12.44
	38	12	"	"	"	"	12.25
	38	12	"	"	"	"	12.20
	38	12	"	"	"	"	12.15
	38	12	"	"	"	"	11.96
	38	12	"	"	"	"	9.48
F	38	12	"	"	" *	"	3.92
G	43	17	"	"	"	"	3.14
	48	22	"	"	"	"	2.55

The percentages of nitrogen, calculated on water-free casein, as before, are stated in detail in Table II., and are concisely recapitulated in Table III.

TABLE III.—*Recapitulation of Results of Nitrogen Determinations by Soda-lime under Different Conditions.*

Series.	Soda-lime.		Packing.	Time of combustion.	Nitrogen found.			Average loss of Nitrogen in per cent. of total Nitrogen.
	Anterior layer.				Maximum.	Minimum.	Average.	
	Kind.	Length.						
A	Coarse.	cm. 12	No channel.	$\frac{3}{4}$ hour.	per cent. 12.44	per cent. 12.43	per cent. 12.43	per cent. . .
B	Fine.	12	Channel.	"	12.25	11.66	11.92	3.3
C	"	7 $\frac{1}{2}$	"	"	12.38	10.51	11.63	6.4
D	Coarse.	12	No channel.	2 $\frac{1}{3}$ hours.	12.27	11.96	12.08	2.8
E	Fine.	12	Channel.	"	12.44	9.48	11.75	5.5
F	"	12	"	" †	one	det'n.	3.92	68.5
G	"	†	"	"	3.14	2.55	2.85	77.0

\* At very high heat.

† 17 and 22 cm.

‡ At very high heat.



It thus appears that in the experiments of Tables I. and II. :—

When the determinations were made with the precautions named, series A, the duplicate results agreed as closely with each other and with those by the Kjeldahl method as could be desired. This, taken in connection with the facts reported in other papers of this series, constitutes strong evidence of the correctness of the figures for percentages of nitrogen.

In series B a channel was left over the soda-lime in the tube, the other conditions were the same as in A. The percentages of nitrogen fell to an average of 11.92. Taking 12.43, the average of the results obtained in the series A and by the Kjeldahl method, as actual per cent. of nitrogen, this makes a loss of 0.4 per cent. of the weight of the water-free casein, or 3.4 per cent. of the total nitrogen.

Series C was a duplicate of B, except that the anterior layer consisted of fine particles of soda-lime, thus offering slightly less surface for contact of volatile nitrogenous products with the latter. The percentages of nitrogen were slightly less than in B. The average was 0.8 short of 12.43 per cent., which makes a loss of 6.4 per cent. of the total nitrogen. That the loss in this series averaged so much more than in the previous one may be largely accidental. The loss in the individual determinations ranges from 0.4 to 15.4 per cent. of the total nitrogen. Both the small amounts of nitrogen obtained and the wide variations in the results bear witness to the error introduced by leaving the channel in the tube. It seems to us most probable that this loss is mainly due to incomplete ammonification of nitrogenous decomposition products. In series A, in which the tube was closely packed with soda-lime, these latter were brought more into contact with the heated soda-lime, i.e., with heated water-vapour, and their nitrogen was completely changed to ammonia. In series B and C, in which there was considerable open space in the tube, the contact with soda-lime would be less intimate, and the ammonification might on that account be less complete. At the same time it is possible that the slower current of gas, consequent upon the larger open space, might, by exposing the ammonia for a longer time to the heat, lead to dissociation.

(To be continued.)

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

AN IMPROVED BURETTE. L. L. DE KONINCK. *Zeitschr. f. angew. Chemie.*—In the estimation of sugar, volumetrically with Fehling's solution, or in the volumetric estimation of carbonates, or, in fact, in any analysis, in which heat is employed, with a standard solution from a burette, it is well known that error is likely to occur from the rising of the steam, which warms the burette, and therefore causes expansion, and consequently inaccuracy. The author has devised a plan by which this error is removed. An ordinary burette is taken, and is attached as usual to a stand, which must be made of iron. To the lower part of the burette a piece of india-rubber tubing is attached, and to this a piece of glass tubing, which projects downwards for about two inches, and then is bent at a right angle, away from the burette and towards the back of the stand, for about five or six inches, and the end of this tube is turned downwards, the whole

length of the glass tubing being about eight inches. Beneath this, fixed to the iron rod of the stand, is an ordinary retort ring, with a screw so that it may be moved up and down as required, and which will hold the dish or other vessel in which the analysis is performed. A Bunsen burner is all that is now necessary for the heating of the liquid, from which the steam rises, but, as will be easily comprehended, only comes in contact with the extreme end of the glass tube attached to the burette, and therefore no error can possibly accrue from expansion either of liquid or burette. To the iron rod of the stand is attached a clamp, the end of which grasps the india-rubber tubing, by means of which regulation in the delivering of the solution is effected. The clamp is controlled by a spring and a screw.

L. DE K.

ESTIMATION OF ORGANIC AND AMMONIACAL NITROGEN RESPECTIVELY IN MANURES.  
M. SAUGERON.—*Arch. de Pharm.*, 3, 1.—The author uses Schauffler's apparatus for this



purpose, which he applies as follows:—As this apparatus was not figured in the ANALYST at the time of its introduction, we take the opportunity of the present abstract to describe it, although it is probably already familiar to several of our readers. In the illustration A is a burette to contain the reagent used, which is a solution of sodium hypobromite, prepared by dissolving 5 grammes of bromine in 50 grammes of solution of sodium hydrate (specific gravity 1.33) and adding 100 grammes of water.

B is a flask to contain the substance under analysis. The cork that closes the flask B has three tubes, *a*, *b*, *c*. The tube *a* terminates in a cock *r*, leading to the external air. The tube *b* extends from the burette to the flask, and can be closed by a pinchcock *p*. The tube *c* puts the flask in connection with another burette *C*, in which the evolved gas is measured. This latter has a reservoir *R* attached, which can be raised or lowered at pleasure, just as in an ordinary nitrometer.

The reaction involved is the well-known action of the hypobromite in causing ammoniacal salts to give up all their nitrogen.

*Estimation of the Ammoniacal Nitrogen.*—1 to 5 grammes of the sample (according to richness) is reduced to as fine a powder as possible, and two equal parts are weighed out, one being used for the ammoniacal and the other for the organic nitrogen. The burette A is charged with the reagent, and one of the weighed portions is introduced into the flask B. The tap *r* having been opened, the reservoir R is raised until the level of the water in the measuring tube rises to 0, and then the tap is closed. The reservoir having been taken in the right hand, the left is applied to the pinch-cock *p*, so as to cause the reagent to flow into the flask in small quantities at a time. As the gas is evolved the reservoir is continually lowered to avoid putting any pressure on the gas and thus reduce the chances of its solubility in the water in the measuring tube. It is necessary to agitate the flask to aid the action, care being taken to hold it by the lip



to avoid warming the gas. When the addition of a further quantity of the reagent causes no more evolution of gas, the operation is terminated and the reservoir is hung up on the catch. At the termination of say five minutes, when the gas has assumed the temperature of the room, the level is adjusted and the gas is measured, and, having been corrected from volume to standard temperature and pressure, it is calculated from volume to weight. It is always advisable to do another estimation on a similar amount of the manure after drying, so as to find the quantity of ammonia dissipated by evaporation and consequently free.

*Estimation of the Organic Ammonia.*—The weighed portion of the sample saved for this purpose is placed in a balloon flask having a neck at least four inches long; 15 c.c. of fuming sulphuric acid are then added, and the flask having been placed in an inclined position to avoid spurling, the whole is gently heated until it ceases to froth. The temperature is then increased until the acid boils, and it is maintained at the boiling point for two hours. The flask is now and then turned upon its axis to bring any carbonised particles that may spurt up, once more within the action of the acid. When all solid particles have disappeared and the liquid has assumed a translucent yellowish or brownish appearance, 2 or 3 decigrams. of powdered potassium permanganate are very cautiously sprinkled in, and the whole is allowed to cool. When cold the contents of the balloon are rinsed into 250 c.c. of distilled water, neutralised with soda so as to leave the liquid just distinctly alkaline, and finally the whole is diluted up to 500 c.c. with water. During these last operations the whole must be kept cold and in a closed vessel to avoid the loss of ammonia. The organic nitrogen having been thus converted into ammonium sulphate, 5 to 10 c.c. are taken and treated in the Schaudler's apparatus with the hypobromite, and the nitrogen estimated. The calculations are evident, and the author having tested the results against those obtained by combustion, finds them very satisfactory. The process is stated to be very simple in practice and to go on with very little attention.

W. H. D.

*ESTIMATION OF DISSOLVED CARBONIC ACID.* L. VIGNERON. *Comptes Rendus*, December, 1887.—On adding a few drops of a pure solution of phenolphthalein to lime water, we obtain a coloured liquid that rapidly becomes colourless on passing carbonic acid or on presenting it in the shape of a solution by the help of carbonate of lime. This reaction is employed by the author for the determination of the carbonic acid existing in natural waters other than that present as normal carbonates. The operation consists in adding phenolphthalein to the sample, and then adding standard lime water until a rose tint is obtained. The lime water is standardised with decinormal sulphuric acid and cochineal. It is necessary to do two experiments on equal volumes of the water as follows. One portion of the water is boiled to expel the carbonic acid, and of this 50 c.c. are placed in a tube of 3 centimetres in diameter with 10 drops of saturated solution of phenolphthalein and the standard lime water is added until a slight tint is produced. The amount required is noted and the liquid preserved for comparison as shown below. This done, 50 c.c. of the water are measured out and mixed with 10 c.c. of phenolphthalein as before, and titrated with the standard lime with constant shaking, and resting between each addition of the reagent until the last addition fails to cause any lessening in colour. At the finish of the process, it is necessary that the colour obtained

should exactly match in the tube reserved from the former experiment for the purpose of this comparison. The colours being thus identical we may properly assume that both tubes contain an equal quantity of free lime, and that the difference between the quantity of lime used in the two experiments will be equivalent to the amount of the dissolved carbonic acid present. The calculation is obvious. The presence of chlorides, nitrates or sulphates of calcium and magnesium does not affect the process. If the boiled water shows alkalinity on the addition of the phenolphthalein it is evident that the water is one containing alkaline salts or magnesium carbonate, and in this case it is necessary to add to the sample (before titrating) a sufficient quantity of pure sodium chloride, to transform such salts into chlorides. In waters containing much carbonic acid the addition of the lime water will cause a very marked precipitate, but in this case we may either wait till it has settled clear, or we may render the comparison liquid equally turbid by the addition of a pinch of pure calcium carbonate. The author claims that this process will estimate as little as 1 c.c. of carbonic acid in a litre of water.

W. H. D.

SEPARATION OF GOLD, PLATINUM, TIN, ANTIMONY, AND ARSENIC. L. DE KONINCK AND A. LE CRENIER. *Rev. Univ., Zeitschr. f. angew. Chemie*, No. 9.—The quantitative separation of these metals, one of the most troublesome in mineral analysis, has been greatly simplified by the authors. The mixed precipitated sulphides are dried and placed in a porcelain boat, which is then put inside a combustion tube. The end of this tube is bent and connected with a Volhard's bulb apparatus, filled with dilute hydrochloric acid. Hydrochloric acid gas is now slowly passed through the tube, which is gradually heated to redness. The tin and antimony volatilise as chlorides and are dissolved in the liquid in the bulbs, whilst the arsenic will be found in the fluid as insoluble sulphide, which may be at once filtered off and weighed. Gold and platinum are left in the boat, and are separated by the usual process.

The authors prefer to evolve the hydrochloric acid from sulphuric acid and lumps of ammoniac chloride.

L. DE K.

POLARISATION TUBES. M. MÜLLER. *Zeitschr. f. angew. Chemie*, No. 9.—Tubes made of metal are liable to sudden variation in length, which may cause an error of about 1 per cent. of sugar. Glass tubes do not suffer from this inconvenience, but as the author found to his cost they often crack from apparently no cause. Tubes which have been allowed to cool, whilst transmitting through them a current of air are the most reliable, but as there is after all no dependence to be placed in them, the author has, in his laboratory, replaced them by porcelain ones, which give every satisfaction and besides look very handsome.

L. DE K.

VOLUMETRIC ESTIMATION OF ANTIMONIC ACID. A. YOLLES. *Zeitschr. f. angew. Chemie*, No. 9.—The author, a short time ago, published a convenient process for the estimation of antimonious acid by means of alkaline potassic manganate and has now extended it to antimonie acids or antimonates. About 0.5 gm. of the compound is put into a 250 c.c. flask and dissolved in a sufficiency of hydrochloric acid. A strong solution of sodic sulphite is gradually added and the liquid digested for some time. The excess of sulphurous acid is now boiled off, and, after cooling, the liquid is made up to the mark, when an aliquot part is taken, and titrated with the manganate. As the liquid contains some free acid, care must be taken that the latter contains sufficient alkali. L. DE K.



TEST FOR "HYDRONAPHTHOL."—Mr. Beam, of Philadelphia, in repeating this test (ANALYST, No. 143, p. 52), has observed the peculiar fact that when the test is performed in strong sunlight, the colour does not appear, but will be produced by placing the liquid in the dark. The coloured liquid will be again bleached in the light, and the colour again restored in the dark; and these changes may be repeated several times.

W. H. D.

DETECTION OF DRIED BLOOD-STAINS. F. DE LA BELLONE. *Arch. de Pharm.*, 3, 115.—If the stains be on cloth, the latter is cut up into very narrow slips, and the threads having been torn apart by means of a needle, they are placed in a very fine glass tube in the surface of a solution of sodium chloride (1 gm. per litre). At the end of several hours the liquid will take a brownish-rose tint, and should then be examined by the spectroscope for the hemoglobine absorption bands. This preliminary test having been made (and it is very valuable), the presence of blood discs is to be assured as follows:—The solution is to be treated with a drop of a concentrated solution of chloral, the rose-coloured precipitate thus produced is allowed to settle and the supernatant liquid is removed with a pipette. A drop of the precipitate is placed on a microscopic slide and gently warmed in the flame of a spirit lamp. This causes it to form a coagulum, from which the liquid is removed by blotting-paper. The coagulum remaining on the slide is now stained with a solution of fuschine, and, when the stain has fairly taken, the excess of dye is washed off with water. A drop of acetic acid is now placed on the slide and a covering-glass put on. The preparation becomes transparent and the fuschine fixes itself on the blood discs, so that they cannot be mistaken when viewed under the microscope. If the stains be found on instruments, on wood, or on stone, the stained portion is to be scraped, and the powder is to be placed on a little piece of cambric, tied up, and coated on the solution of salt. In cases where we have to deal with earth, it is necessary to put it under a dissecting microscope and to separate the fragments that appear to be stained. These are then placed in the little cambric bag and treated as already described.

W. H. D.

#### LAW NOTES. TO OUR READERS.

Some years ago it was decided to discontinue the reporting of ordinary police-court proceedings, and only to give such cases as authoritatively established some point in connection with the working of the Acts which public analysts are interested. After a fair trial of this system, a majority of the members of our Society have expressed a wish that our old practice should be, to some extent, returned to, and, in reference to such request, we have decided to resume the reporting of police proceedings. The line will be drawn at accounts of ordinary cases, and reports will only be inserted when any novel, legal, or chemical point arises, or where the certificate of the analyst is in any way attacked. Any member or subscriber connected with such a case is, therefore, invited to furnish us with a report of the proceedings, signed with his name, not for publication, but as a guarantee of exactitude.

ALUM IN BREAD—IMPORTANT DECISION.—In a Divisional Court of Queen's Bench, on April 9th, an important point was decided under the Food and Drugs Act. A baker was prosecuted by the Local Authority of Nottingham, for selling bread adulterated with alum. The baker pleaded that he had not put alum into the bread, and if it were there it might have been in the flour when he bought it, and it was not in the flour to his knowledge. The Justices dismissed the case, holding that he was not liable, as he did not know of the presence of the alum.

Their Lordships now reversed the judgment of the Justices, holding that knowledge of the adulteration on the part of the seller is not necessary.

THE CONSTITUENTS OF "NEW MILK."—At the Bolton Borough Police Court, on the 2nd ult., Alice Heaton, of 130, Blackburn Road, was summoned at the instance of Inspector Maguire, for having sold, to the prejudice of the purchaser, quantities of milk which were not of the nature, substance, and quality of the article demanded on the 28th March.

The Town Clerk (Mr. R. G. Hinnell) prosecuted, and Mr. Fielding appeared for the defence.

In opening, Mr. Hinnell explained that there were two charges against the defendant, who keeps a milk-house at the address given. A person who was living with her, and reputed to be her husband, was summoned on a similar charge about a week ago, but was discharged on the statement that his wife was the tenant, and conducted the house. The present defendant was a middle trader, and in addition to selling milk at the house, disposed of it by means of boys who went about with cans, and were paid at the rate of 3d. in the shilling at the week end. On the date in question the inspector purchased a pint of "new milk" from a boy named Hill, for which he paid 1½d. The youngster stated that he was selling for Heaton's. The inspector then proceeded to the defendant's premises at 7 a.m., where he purchased a similar quantity from the husband. The samples were analysed and found to be so adulterated that he applied for the imposition of a heavy penalty.

Mr. Fielding raised a technical objection on the ground that the summons should have been served at a period not exceeding twenty-eight days from the time the offence was said to have been committed—the time when the milk was purchased. He submitted that twenty-nine days had intervened.

Colonel Winder (the Magistrate's Clerk) said the section read "not exceeding twenty-eight days from the time of the purchase." By seven o'clock, when the purchase was made, part of the day had gone. The 28th did not count; the time only began to run at midnight on the 28th.

Mr. Fielding pointed out that by the penal code when a man was committed for a month the sentence began to operate immediately after the committal.

Alderman Nicholson said the objection would be overruled.

Evidence having been called by the Town Clerk as to the tenancy of the house and the payment of rates,

Inspector Maguire was again brought into the witness-box, and produced the register of milk dealers. An entry in the book gave "John Heaton" (defendant's husband) as the registered owner.

The result of the analysis of the milk was submitted by the Town Clerk.

The analyst (Dr. Sergeant) found that there was a large quantity of starch in the sample purchased from the boy. There were 59 parts of milk to 41 parts of water, and the "milk" was thickened with added starch, coloured probably with treacle. The latter statement also applied to the second sample, which contained 58 parts of milk to 42 parts of water.

Mr. Fielding submitted, in defence, that the boy Hill was not and never had been a servant of the defendant; he could sell the milk, how, when, and where he liked, and was paid on commission. If he spilled the milk he would have to bear the loss himself. With regard to the second case, he contended that the defendant was acting simply under instructions from her husband.

The magistrates retired for a short time to consider their decision, and on re-entering the court announced that the case in the first charge would be dismissed, as they believed the boy had acted as agent, and not as servant. They convicted in the remaining one, the only question in this case being as to the amount of the fine, as the adulteration had been very serious. A penalty of 40s. and costs would be imposed, or an alternative of 14 days' imprisonment.

**WHAT IS BRANDY?—APPLICATION FOR A MANDAMUS.**—On the 13th ult. Mr. Forrest Fulton applied to Mr. Justice Field and Mr. Justice Wills for a rule for a *mandamus* to compel a Bench of Justices at Lindsey, in Lincolnshire, to state a case for the opinion of their lordships.

A superintendent and a sergeant of police had gone to Mr. Rowe, a publican, and asked for a pint of brandy. What they got was British brandy, and the magistrates convicted the publican, under the Adulteration Act, of having sold an article which had not been demanded by the purchaser. It was now submitted to their lordships that the article sold was well-known as brandy, and therefore the conviction was not a proper one. It was not only brandy, but it was believed to be more wholesome than French brandy. Mr. Justice Field said that the question whether the article was brandy was one of fact for the justices, and there seemed to be no question of law for them. The application was refused.

**HOW ANALYSTS DIFFER.**—At Barton-on-Humber police-court a case of a remarkable character was decided last month. On the 16th April last, Joseph Reynolds, of the Swan Inn, Barton, was summoned for selling to Police-inspector Ward a pint of liquor, which was not of the nature, substance, and quality of the article demanded. The sale having been proved, the certificate of Dr. Graham, of University College, London, the Public Analyst for Lincolnshire, giving the result of his analysis, was put in. This certificate stated that the liquor was not brandy, but alcohol coloured and flavoured to imitate brandy, and that it contained 76½ degrees of proof spirit.

Mr. Sowter, of Brigg, who appeared on behalf of Messrs. Sutton and Co., brewers, etc., Brigg, the owners of the house, applied for an adjournment, which was granted, and in the meantime a third sample of the liquor, in the hands of the police, was ordered to be sent to Somerset House for analysis. When the case came before the magistrates the other day the clerk to the justices read the certificate of analysis which he had received from Somerset House, and which was to the effect that Dr. J. Bell, Dr. R. Bannister, and Dr. Helm certified that the liquor contained 80 per cent. of proof spirit, and that it was brandy.

The case was at once dismissed.

Mr. Sowter said had it been necessary he was quite prepared to prove that the liquor was Cognac brandy, imported from Charente,



# THE ANALYST.

JULY, 1888.

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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

Ordinary meeting of the Society was held at Burlington House, Piccadilly, on Tuesday, the 20th June, the President, Mr. A. H. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened, the following gentlemen were declared to be elected:—

As Members—J. Bemrose, analytical chemist, Montreal; F. L. Teed, D.Sc., F.I.C. S., public analyst for Islington; W. F. Lowe, F.C.S., F.I.C., public analyst for ... As Associates—W. D. Mildred, assistant to Mr. A. C. Wilson; F. W. ... assistant to Mr. Rimmington.

The following papers were read and discussed:—

"Polluted Drinking Water and the Closure of Wells." By Dr. Alfred Hill.

"On the Adulteration of Lard with Cotton Seed Oil." By Mr. A. H. Allen.

" " " " By Mr. Otto Hehner.

" " " " By Mr. E. W. T. Jones.

" " " " By Mr. R. Williams.

"On the Temperature of Ether in the Soxhlet Extractor." By Dr. P. Vieth.

"Notes on Senna." By C. Heisch.

"Further Note on Pepper Analysis." By C. Heisch.

At the conclusion of the discussion on Dr. Hill's paper, Mr. Otto Hehner proposed, Bostock Hill seconded, and it was unanimously resolved,

"That a deputation from the Society of Medical Officers of Health and the Society of Public Analysts wait upon the President of the Local Government Board,

and urge upon him the necessity of altering the wording of the 70th section, 'injurious to health,' to 'dangerous to health.'"

The next meeting of the Society will be held at Sheffield on August 10th. Full particulars will be duly announced.

#### ON THE WEAR AND TEAR OF PLATINUM DISHES.

By DR. P. VIETH, F.C.S., F.I.C.

(*Read at the Meeting, May, 1888.*)

It would be more than superfluous in this place to enumerate and dwell at length upon all the causes by which platinum vessels used for analytical operations are deteriorated; suffice it to say that the most noteworthy are the kind of operations for, and the frequency with which the vessels are used, and the treatment they receive while in actual use, and—a not at all unimportant point—when being cleaned.

The platinum dishes to which the following remarks refer have a rounded bottom; their internal diameter is  $1\frac{1}{2}$  in., their greatest depth  $\frac{1}{2}$  in. The edge is pressed round so as to form a flat rim,  $\frac{1}{8}$  in. wide; on one side the rim is more than double this width, allowing to better handle the dishes, and, moreover, offering space to stamp them with a number. The weight of the dishes varies from ten to fourteen grammes.

The dishes are employed almost exclusively for determinations of solids and ash in milk and milk products, and used in rotation as regular as possible.

In November, 1880, I started work with sixty of these dishes, but two months later found it necessary to add another twenty, and in January, 1886, increased the number to one hundred. As the number of samples analysed is about twenty thousand a year, every dish is used on average four times a week.

There are from 250 to 300 samples every year of which more full, and in a great many instances, complete analyses are made with every care and precaution. These analyses are invariably done in duplicate, and the dishes in these instances are ignited and specially weighed before use. With the bulk of the samples taken merely for controlling purposes, I am obliged to deal in a more expeditious manner, and limit myself to the gravimetric determination of the solids, besides ascertaining the specific gravity. The dishes are charged with 5 grms. of milk, kept for three hours on a steam-bath, for other three hours in an air-bath at  $100^{\circ}$  C., and weighed. They are then put in hot water, in which some washing soda has been dissolved, and kept therein for about one half hour. After that time it is found that the milk residue is so much softened that it can be brushed out without difficulty by means of a bass-brush, the material of which is, I believe, furnished by the dried stalks of some kind of a sedge. The dishes are next transferred into a fresh hot soda solution, and the remaining traces of milk solids brushed off. Rinsing in water, and carefully wiping with a duster, finish the cleaning operation, and leave the dishes ready for renewed use. After various other modes of procedure had been tried, the manner of cleaning just described has been found to answer the purpose best, and tell upon the weight of the dishes very insignificantly indeed.

The only spot where I have found some of my dishes exhibit marks of wear is the inner edge, *i.e.*, that part where the platinum is sharply bent to form the rim, and



French, at 35s. (contained wheat, or barley, or perhaps rye starch) ...	11.02	4.62	.36	.19	.66	.16	.24	.52	2.22
French, at 40s. (contained potato starch) ...	10.72	4.52	.82	.24	.40	.66	.30	.58	1.18
French, (contained added starch) ...	11.15	3.44	.32	.28	.42	.04	.28	.40	1.24
French, (contained added starch) ...	10.05	7.70	.37	.09	.63	.10	.27	.75	5.06
Smyrna (block), at 42s.	17.74	8.16	2.57	.46	.68	1.92	.34	.48	.12
Own Make ...	14.66	4.34	1.54	.16	.42	.70	1.04	—	.12

	Percentage of Ash, deducting silica, oxide of iron and alumina.	Percentage composition of Ash, after deducting silica, oxide of iron, and alumina.					
		K <sub>2</sub> O	Na <sub>2</sub> O	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	C <sub>2</sub> , ClO, and Undetermined.
Italian (Solazzi), at 140s. per cwt. ...	4.02	40.5	2.7	14.4	23.1	9.4	9.9
Italian, at 84s. per cwt. ...	4.32	39.8	4.4	26.2	11.5	5.5	12.6
Italian, at 84s. per cwt. ...	5.07	36.8	10.9	10.5	12.2	8.1	21.5
Italian ...	3.08	43.5	3.9	15.9	5.2	11.0	20.5
Italian ...	3.45	40.3	4.0	11.1	7.5	8.4	28.7
Unknown, 75s. to 80s.	4.09	35.0	8.3	21.5	15.6	6.1	13.5
Unknown, 75s. to 80s. (contained some rice starch)	3.90	34.1	6.4	13.8	24.6	8.2	12.9
French, at 35s. (contained wheat or barley, or perhaps rye starch) ...	1.88	19.1	10.1	35.1	8.5	12.9	14.3
French, at 40s. (contained potato starch) ...	2.76	29.7	8.7	15.2	23.9	10.8	11.7
French (contained added starch) ...	1.80	17.8	15.6	23.3	2.2	15.3	25.8
French (contained added starch) ...	1.89	29.6	4.7	33.3	5.3	14.2	12.9
Smyrna (block), at 42s.	7.56	34.0	6.1	9.0	25.4	4.5	21.0
Own Make ...	4.22	36.5	3.8	9.9	16.3	24.7	8.3

The genuine juices contain a tolerably large proportion of starch, which, however, may easily be distinguished under the microscope from most of the starch added for purposes of sophistication.

It will be seen that in the samples in which adulteration was detected by the microscope there was a large drop in the proportion of (silica and clay free) ash, which in the best, and presumably genuine samples ranges from about 3 to 5 per cent. In three adulterated cases out of four the ash fell below 2 per cent., and in the remaining case below 3.

The figure of chief interest in the composition of the ash is the potash, which constituted from 34 to 43 per cent. of the (silica and clay free) ash in the genuine samples, but was only from 18 to 30 in the adulterated ones.

The sample made by the writer himself agreed well, as regards both the proportion

of ash and its composition, with the commercial samples of reputed genuineness, except that it contained a much larger percentage of phosphoric acid.

Of course the chemistry of the ash is only one side from which the subject may be approached, and the information afforded by it is anything but satisfactory or complete; but it struck the writer that the figures obtained by him might be of service to others who may be called upon to deal with the subject.

There seems to be no doubt at all that a large proportion of the liquorice in the market—notably that from French sources—is grossly adulterated, and, as far as regards such specimens as those referred to, there should be no difficulty in dealing with the matter under the Sale of Food and Drugs Act.

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(Read at Meeting, May, 1888.)

MANY kinds of fat extractors have been from time to time devised, some of which are specially suited for certain cases, but the form which has gained most favour (and deservedly so) for general purposes, is the Soxhlet.

This beautiful piece of apparatus is not, however, faultless, one imperfection being found in the fact that the liquid in contact with the substance to be extracted, particularly if it be one of the less volatile solvents, such as alcohol, is usually at a temperature considerably below its boiling point, to the detriment of its extractive power.

Another weak point is the not infrequent irregularity in the action of the syphon, which will sometimes become filled as to its longer limb, with alternate portions of liquid and vapour, or air, and in consequence empty the tube in which is the substance to be extracted, before the liquid in it has reached its proper level.

These failings led me to design the modification of it, which is here illustrated.

As may be seen in the cut, the essential parts of a Soxhlet, viz, a tube to contain the substance to be exhausted, and a syphon to empty it periodically, are retained.

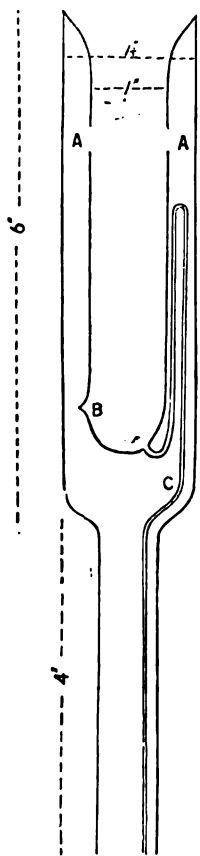
These are, however, enclosed in an outer tube, which is fused to the inner tube at its upper end. The apparatus is attached by cords at its upper and lower ends to a vertical condenser and a flask, respectively, just like a Soxhlet.

The action is then as follows :—

The vapour of the liquid boiling in the flask passes up the annular space between the inner and outer tubes, and flows into the upper part of the inner tube through the holes *AA* (see cut). Thence it goes up into the condenser, and is there cooled, and falls back in drops into the inner tube, which is thus gradually filled; as the inner tube is completely jacketed with the vapour of the solvent, the liquid in it is kept at its boiling point the whole time it is in contact with the substance to be extracted. When the inner tube

is filled to the level of the upper bend of the syphon it is, of course, emptied by it in the ordinary way, and the process of filling again begins.

I venture to think that this apparatus will be found to have several advantages over the ordinary Soxhlet, among which are the following :—





- (1) The solvent is kept at its boiling point in contact with the substance to be extracted.
- (2) The syphon acts with great regularity; I am inclined to attribute this to its being kept at a uniform temperature throughout its length, and to the absence of any constriction, such as occurs in the case of the Soxhlet at the point where it passes through the wall of the lower tube connected with the flask.
- (3) There is less liability of breakage, as it has no projecting parts, but presents a smooth cylindrical surface.

A good condenser with a wide tube should be used with it, as at the moment when the inner tube of the apparatus begins to empty itself, the liquid it contains (being just at its boiling point, and slightly agitated by the action of the syphon) partly vapourises, and thus throws additional work on the condenser.

It is to be noted that the rate at which drops fall from the condenser into the inner tube, does not measure the speed with which the latter is being filled, as a certain proportion of them do not originate from the condensation of the vapour sent up from the flask below, but from the liquid already in the inner tube.

The apparatus is made in two sizes, and can be obtained from Messrs. Townson and Mercer, 89, Bishopsgate Street, E.C., at the same price as an ordinary Soxhlet.

AA are two holes in the inner tube of the apparatus, affording means of communication with the outer tube.

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## THE TEMPERATURE OF ETHER IN THE SOXHLET EXTRACTOR.

By DR. P. VIETH, F.C.S., F.I.C.

(Read at Meeting, June, 1888.)

WHEN, two months ago, Mr. Blount made us acquainted with a new form of fat extraction apparatus, he claimed as one of the advantages of the new apparatus over the original Soxhlet Extractor, that the former allows to carry out the extraction with boiling ether. The question was then, as on similar previous occasions, discussed, whether the ether in the well-known Soxhlet was at or near boiling point, opinions being very much divided. My query whether the opinions expressed were founded on personal observations was in every case but one answered in the negative. While admitting that I had never made the simple experiment myself, I have always concluded that the ether is boiling or near boiling, giving as my authority Professor Soxhlet himself, who is personally known to me as one of the most exact and trustworthy analysts.

However, in order to satisfy myself by an experiment of my own, I made some observations with regard to the point in question. The six Soxhlets which I have hung up in a row side by side were working at the time of the experiment, the thermometer being kept in No. 5. The six flasks are immersed in a common water-bath of rectangular shape, through which a continuous current of hot water can be, and was, conducted. The extraction lasted two hours, during which time eight readings were taken at

Seyfert pretends that the analysis goes wrong if not exactly 50 c.c. of 45 per cent. alcohol are added. In my experiments I gelatinised the starch with what I guessed would be about 100 c.c. of water, so I could not be quite sure about the exact quantity of alcohol, but still the results did agree. If, however, analysts go and *seriously* alter the proportions, or use alcohol of greatly different strength, or let the mixture stand too long, say for 24 hours, they must not expect to get proper results.\*

#### TEST ANALYSES.

- |        |  |
|--------|--|
| No. 1. | 10 c.c. baryta water = 30.15 c.c. acid<br>1.0345 substance taken. Filtrate = 24.75 c.c. acid<br>= 84.56 per cent. of starch. |
| No. 2. | 10 c.c. baryta water = 29.9 c.c. acid<br>1.5635 substance taken. Filtrate = 21.7 c.c. acid<br>= 84.96 per cent. starch.      |
| No. 3. | 10 c.c. baryta water = 29.6 c.c. acid<br>1.587 substance. Filtrate = 21.35 c.c. acid<br>= 84.21 per cent. starch.            |
| No. 4. | 10 c.c. baryta = 29.55 c.c. acid<br>1.600 substance. Filtrate = 21.25 c.c. acid<br>= 84.04 per cent. starch.                 |

The average percentage of starch is therefore 84.44.

The same sample was now analysed in duplicate by Lintner's process (conversion into glucose, and weighing the reduced metallic copper). I obtained 84.06 to 83.98, average 83.87 per cent. of starch.

There is therefore no fault to be found with the process when analysing the pure form of starch. Whether I will be again equally successful when dealing with cereals we will see later on.

In my first paper I pointed out, no satisfactory determination of starch could be arrived at by any method based on inversion. But probably I shall have to modify this opinion in regard to Lintner's process.

#### "HYDRONAPHTHOL" NOT BETANAPHTHOL.

By JUSTUS WOLFF.

In the March edition of the ANALYST appeared a note on a test for hydronaphthol, communicated by Mr. Alfred L. Beebe, assistant chemist of the New York City Health Department, in which he stated that "hydronaphthol," as is well known, is really a trade name for "betanaphthol."

Now I beg to state herewith, that if Mr. Beebe had investigated the nature and properties of both hydronaphthol and betanaphthol, he would have found them to be of different chemical constitution. In chemistry an opinion or assertion that a thing is so amounts to nothing, even if the great Justus von Liebig would have said it; but for all assertions proofs must be produced through facts, reactions, and experiments. Truth has to be found by research, and no assertion can be founded on a phrase such as "as is well known."

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by translator.—Who would think of seriously altering the proportion of the ingredients, eg's solution or Wanklyn's water solutions?—L. DE K.



In order to prove the error of Mr. A. L. Beebe as to his opinion on hydronaphthol, give the following facts and reactions of both hydronaphthol and betanaphthol, which, to any sound mind, will demonstrate that these compounds are of different chemical constitution.

1st. Hydronaphthol is prepared from commercially pure betanaphthol by a process by which the chemical constitution of the latter is changed, and therefore hydronaphthol cannot be betanaphthol.

2nd. The melting-point of hydronaphthol is  $241^{\circ}$  F., whilst that of the betanaphthol from which it is prepared, and of all other betanaphthols, is  $252^{\circ}$  F., another proof for the difference in the chemical constitution of hydronaphthol and betanaphthol.

3rd. In a solution of five grains of hydronaphthol in a fluid drachm of alcohol of 95 per cent. one to two drops of tincture of iron (U. S. P., 1880) produce a dark yellowish-brown coloration, whilst in a solution of the same quantity of betanaphthol, in the same quantity of the same quality of alcohol, with the same quantity and quality of tincture of iron under the same circumstances, a fine green coloration is obtained (Herman Wolf's naphthol test). Here, too, we have another proof that hydronaphthol cannot be betanaphthol.

4th. Hydronaphthol is decomposed at a higher temperature than that at which hydrates give off water, and forms water, whilst betanaphthol, at a still higher temperature, i.e.,  $545^{\circ}$  to  $554^{\circ}$  F., distils undecomposed, which proves that hydronaphthol is the hydro-compound, whilst betanaphthol represents the corresponding anhydro-compound, both of which, of course, cannot have one and the same constitution.

5th. Hydronaphthol dissolves in four to five parts of alcohol with a brown colour, whilst, under the same circumstances, betanaphthol dissolves colourless; another difference between hydronaphthol and betanaphthol.

6th. The antiseptic limit of hydronaphthol is 1:7200, whilst that of betanaphthol is 1:3800, and therefore these compounds must be of a different chemical constitution.

7th. Hydronaphthol is non-poisonous and harmless, whilst, according to such authorities as Kaposi, Neisser, Young, Squire, Rapan, Weingaertner and Bouchardat, betanaphthol, even in small doses, produces poisonous effects and death. This is also a proof that both compounds must be entirely different one from another.

The conclusions to be drawn from the above facts are:—

Hydronaphthol, being prepared from betanaphthol by a process by which the chemical constitution of the latter is changed, the melting point lowered by  $11^{\circ}$  F., the coloration with iron chloride in alcoholic solution changed from bright green to dark brownish-yellow, the product obtained decomposed at a higher temperature under formation of water (whilst betanaphthol distils undecomposed at a still higher temperature), and dissolves in alcohol with a brown colour as compared with the colourless solution of betanaphthol—it cannot be identical with the latter, but must possess a different chemical constitution.

Further, hydronaphthol possessing an antiseptic limit of 1:7200, whilst betanaphthol has a limit of 1:3800, and the former being non-poisonous and harmless, whilst the latter is a strong and even deadly poison—it cannot be identical with the latter, but must possess a different constitution; nor is it possible that it

of ash and its composition, with the commercial samples of reputed genuineness, except that it contained a much larger percentage of phosphoric acid.

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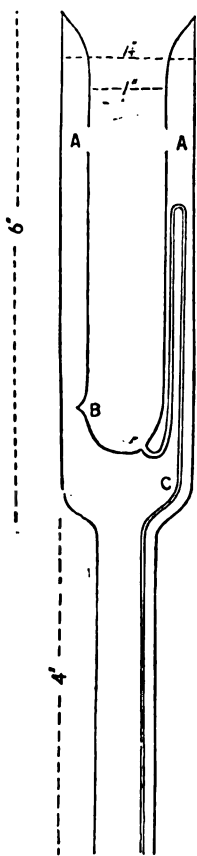
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regular intervals. The observations are subjoined in a tabulated form. From the figures it will be seen that the quantity of ether evaporated and condensed again varied with the temperature of the water in the bath. The temperature of the condensed ether, on the other hand, showed no variations, the thermometer indicating, and remaining stationary at, 35° C. from the time the first drop of condensed ether fell down until the experiment was terminated. It made no difference whether the thermometer bulb was so placed that it was surrounded by ether steam only, or partly or wholly immersed in fluid ether. The thermometer used has a scale extending from - 25 to + 360° C., and the divisions are far too narrow to allow to read off fractions of a degree with anything like accuracy. For my purpose it was sufficient to prove that under the usual conditions under which Soxhlet Extractors are worked, the fat extraction is effected by, practically speaking, boiling ether.

Temperature °C.		Number of drops of condensed Ether per minute.
Water in Bath.	Ether in Extractor.	
55	35	140
51	35	124
50	35	102
67	35	200
59	35	180
52	35	120
57	35	168
56	35	174

*(Conclusion of Society's Proceedings.)*

#### ON THE ESTIMATION OF STARCH.

BY PROF. DR. ALEX. V. ASBÓTH.

MANY analysts have recently investigated my process for the determination of starch, and several of them have given an unfavourable opinion on it.

Of Dr. Monheim's criticism I understand this to be the essence :—Of great influence on the results by v. Asbóth's process is the strength of the baryta solution. With the same solution one can certainly obtain concordant results with the same sample of a cereal, but surely if a sample of maize yields 77·76 per cent. of starch (a quantity hitherto never obtained), whilst the same sample analysed by Lintner's process, by two independent analysts, only gave 65·77 to 65·89 per cent., there must be something wrong. If, however, a baryta solution of other strength is used, the process fails altogether. If, perhaps, my baryta solution was not exactly of the prescribed strength, this was caused through some of the baryta crystallising out, etc.

Friedrich Seyfert writes about as follows :—The sample to be assayed has to be gelatinised as directed, with about 150 c.c. of water. 50 c.c. of standard baryta water are now added, and finally 45 per cent. alcohol up to the 250 c.c. mark. The excess of



baryta is then estimated in the clear fluid. It could not be denied the analysis gave a much lower result, if less than 50 c.c. of spirit were added, and higher results if the amount was increased. Again, on adding more spirit to the clear fluid, a further opalescent precipitate was obtained. . . .

The samples of potato-flour were now again analysed, only 125 c.c. of water being added. When adding the alcohol 50 c.c. of a 99 per cent. spirit were first added, and the remaining space filled up with alcohol of 46 per cent. The liquid now took 1 c.c. less acid, which made the percentage of starch impossibly high, etc.

As neither Monheim nor Seyfert appear to have followed my directions, and have, in consequence, got indifferent results, I think it advisable to give a recapitulation of my process.

For my recent experiments I used maize starch, which was first washed with weak starch, then with weak hydrochloric acid, then with water, and finally with alcohol and ether. On analysis it gave .096 per cent. of ash, .253 of matter insoluble in hot dilute acid, and 13.43 per cent. of moisture. From 1 to 1.6 gramme is repeatedly rubbed in a glass mortar with water, and the milky fluid transferred to a 250 c.c. flask. When all the starch is in, enough water is added to bring the volume up to about 100 c.c., and the flask is then placed in boiling water for half an hour to cause the starch to gelatinise. Frequent stirring is advisable to prevent lumps of starch paste forming. After cooling to the temperature of the room, 50 c.c. of standard baryta water\* are delivered from a special burette, the flask corked and well shaken. Alcohol of 45 per cent. is then added up to the mark, the mixture well shaken, and allowed to settle. This interval I use for standardising the baryta water. 50 c.c. of thoroughly boiled distilled water are put into the flask, 10 c.c. of baryta water are added, and then titrated with deci-normal hydrochloric acid, with phenol-phthalein as indicator. About 30 c.c. of acid are required. If a flocculent precipitate refuses to settle, I filter through glass-wool, but on no account through paper or cotton-wool, as this would cause a loss of baryta and give a too high percentage of starch. 50 c.c. of the filtrate are now titrated. The difference in c.c.'s of acid between the two determinations is first multiplied by five and then by .0324 = starch in quantity taken for analysis.

When Monheim complains of the process not working when baryta water of different strength is used, I must say he is quite right. But I directed to use baryta water of 10ths normal strength (a saturated solution).

It must not be forgotten, the baryta starch is only insoluble when excess of baryta is present. I do not agree with Spence,\* who found the baryta starch to vary in composition. When I investigated its nature I did not always use the same proportion of baryta water, but, providing I added an excess, the composition of the precipitate was always the same.

The baryta solution, after being kept for a long time, certainly loses in strength, but not to such an extent as would render the results inaccurate. In March, 1887, I prepared a solution which required for neutralisation 30.3 c.c. of  $\frac{N}{10}$  HCl, and now, after twelve months, it takes 29.5 c.c. of acid.

\* See *Analyst*, July, 1887, p. 139.

† *Journ. Soc. Chem. Ind.*, 1888, p. 77.

Seyfert pretends that the analysis goes wrong if not exactly 50 c.c. of 45 per cent. alcohol are added. In my experiments I gelatinised the starch with what I guessed would be about 100 c.c. of water, so I could not be quite sure about the exact quantity of alcohol, but still the results did agree. If, however, analysts go and *seriously* alter the proportions, or use alcohol of greatly different strength, or let the mixture stand too long, say for 24 hours, they must not expect to get proper results.\*

#### TEST ANALYSES.

- |        |  |
|--------|--|
| No. 1. | 10 c.c. baryta water = 30.15 c.c. acid<br>1.0345 substance taken. Filtrate = 24.75 c.c. acid<br>= 84.56 per cent. of starch. |
| No. 2. | 10 c.c. baryta water = 29.9 c.c. acid<br>1.5635 substance taken. Filtrate = 21.7 c.c. acid<br>= 84.96 per cent. starch.      |
| No. 3. | 10 c.c. baryta water = 29.6 c.c. acid<br>1.587 substance. Filtrate = 21.35 c.c. acid<br>= 84.21 per cent. starch.            |
| No. 4. | 10 c.c. baryta = 29.55 c.c. acid<br>1.600 substance. Filtrate = 21.25 c.c. acid<br>= 84.04 per cent. starch.                 |

The average percentage of starch is therefore 84.44.

The same sample was now analysed in duplicate by Lintner's process (conversion into glucose, and weighing the reduced metallic copper). I obtained 84.06 to 83.68, average 83.87 per cent. of starch.

There is therefore no fault to be found with the process when analysing the purer form of starch. Whether I will be again equally successful when dealing with cereals we will see later on.

In my first paper I pointed out, no satisfactory determination of starch could be arrived at by any method based on inversion. But probably I shall have to modify this opinion in regard to Lintner's process.

#### "HYDRONAPHTHOL" NOT BETANAPHTHOL.

BY JUSTUS WOLFF.

IN the March edition of the ANALYST appeared a note on a test for hydronaphthol, communicated by Mr. Alfred L. Beebe, assistant chemist of the New York City Health Department, in which he stated that "hydronaphthol," as is well known, is really a trade name for "betanaphthol."

Now I beg to state herewith, that if Mr. Beebe had investigated the nature and properties of both hydronaphthol and betanaphthol, he would have found them to be of different chemical constitution. In chemistry an opinion or assertion that a thing is so amounts to nothing, even if the great Justus von Liebig would have said it; but for all assertions proofs must be produced through facts, reactions, and experiments. Truth has to be found by research, and no assertion can be founded on a phrase such as "as is well known."

\* Note by translator.—Who would think of seriously altering the proportion of the ingredients, say in Fehling's solution or Wanklyn's water solutions?—L. DE K.



In order to prove the error of Mr. A. L. Beebe as to his opinion on hydronaphthol, I give the following facts and reactions of both hydronaphthol and betanaphthol, which, to any sound mind, will demonstrate that these compounds are of different chemical constitution.

1st. Hydronaphthol is prepared from commercially pure betanaphthol by a process by which the chemical constitution of the latter is changed, and therefore hydronaphthol cannot be betanaphthol.

2nd. The melting-point of hydronaphthol is  $241^{\circ}$  F., whilst that of the betanaphthol from which it is prepared, and of all other betanaphthols, is  $252^{\circ}$  F., another proof for the difference in the chemical constitution of hydronaphthol and betanaphthol.

3rd. In a solution of five grains of hydronaphthol in a fluid drachm of alcohol of 95 per cent. one to two drops of tincture of iron (U. S. P., 1880) produce a dark yellowish-brown coloration, whilst in a solution of the same quantity of betanaphthol, in the same quantity of the same quality of alcohol, with the same quantity and quality of tincture of iron under the same circumstances, a fine green coloration is obtained (Herman Wolff's naphthol test). Here, too, we have another proof that hydronaphthol cannot be betanaphthol.

4th. Hydronaphthol is decomposed at a higher temperature than that at which hydrates give off water, and forms water, whilst betanaphthol, at a still higher temperature, viz.,  $545^{\circ}$  to  $554^{\circ}$  F., distils undecomposed, which proves that hydronaphthol is the hydro-compound, whilst betanaphthol represents the corresponding anhydro-compound, both of which, of course, cannot have one and the same constitution.

5th. Hydronaphthol dissolves in four to five parts of alcohol with a brown colour, whilst, under the same circumstances, betanaphthol dissolves colourless; another difference of hydronaphthol and betanaphthol.

6th. The antiseptic limit of hydronaphthol is 1.7200, whilst that of betanaphthol is 1.3800, and therefore these compounds must be of a different chemical constitution.

7th. Hydronaphthol is non-poisonous and harmless, whilst, according to such authorities as Kaposi, Neisser, Young, Squire, Rapan, Weingaertner and Boucharlat, betanaphthol, even in small doses, produces poisonous effects and death. This is also a proof that both compounds must be entirely different one from another.

The conclusions to be drawn from the above facts are:—

Hydronaphthol, being prepared from betanaphthol by a process by which the chemical constitution of the latter is changed, the melting point lowered by  $11^{\circ}$  F., the coloration with iron chloride in alcoholic solution changed from bright green to dark brownish-yellow, the product obtained decomposed at a higher temperature under formation of water (whilst betanaphthol distils undecomposed at a still higher temperature), and dissolves in alcohol with a brown colour as compared with the colourless solution of betanaphthol—it cannot be identical with the latter, but must possess a different chemical constitution.

Further, hydronaphthol possessing an antiseptic limit of 1.7200, and betanaphthol one of 1.3800, and the former being non-poisonous and harmless, whilst the latter is a strong and even deadly poison—it cannot be identical with betanaphthol, but must be of different constitution; nor is it possible that it can be impure betanaphthol, as in that

case its antiseptic limit would be even less than that of betanaphthol, and it would then be much more poisonous than the latter, or at least as strong a poison, whilst it is absolutely non-poisonous and harmless.

The test with nitrous acid or fuming nitric acid, which Mr. Alfred L. Beebe discovered and described, indicates hydronaphthol as well as betanaphthol, both having this reaction in common; it is, therefore, not a distinguishing test, but an indicative one for both.

The above-described tests are some of the distinguishing ones, of which many more are existing, but those mentioned alone prove the different constitution of hydronaphthol and betanaphthol beyond all doubt.

#### ON CERTAIN SOURCES OF LOSS IN THE DETERMINATION OF NITROGEN BY SODA-LIME.

By W. O. ATWATER AND E. M. BALL.

*(Continued from page 115.)*

In series D the determinations were made by the usual method, except that the time of combustion was two and a half hours instead of three quarters of an hour. The loss of nitrogen varied from 1.3 to 3.8 per cent., and averaged 2.8 per cent. of the total nitrogen. An article to follow this will give reasons for attributing the larger part of this loss to dissociation of ammonia during its long sojourn in the tube, though it may be partly due to incomplete ammonification. Briefly stated, they are that (1) the longer exposure to heat, which is longer in proportion than the difference in time would imply, would favour dissociation, and (2) there may have been lack of moisture from the anterior layer of soda-lime after the latter had been heated some time, and this lack may have been unfavourable to complete ammonification but favourable to dissociation.

In series E the conditions were the same as in D, except that a channel was left above the soda-lime. The variations in results and the losses of nitrogen were still greater, the latter ranging from 0.0 to 23.7, average 5.5 per cent. of the total nitrogen. For the case in which all or nearly all the nitrogen was obtained we have no satisfactory explanation, and can only state the results as obtained. That the average loss should be greater and the range of variation wider with a channel than without it is easily accounted for, on the ground that the current of outflowing gases would be slower and the ammonia hence exposed for a longer time to the heat by which it is dissociated.

The single determination F differed from those of E in that the heat was as high as the tubes would endure without bursting. The two in series G differed from those of E in the greater length of the anterior layer of soda-lime. The nitrogen lost in F and G ranged from 68.5 to 79.5 per cent., or from two-thirds to three-fourths of the whole. That the lowest results of all should have been obtained with the high heat and very long tubes is easily explained by assuming that the loss of ammonia by dissociation increased with increase of heat and with increase of time of exposure to heat.

To test the working of the soda-lime method with a substance in which the complete ammonification of the nitrogen is more difficult, a series of trials were made with strychnine. The determinations were made by what has been spoken of as our "usual method," except that—



1st. In several cases extra large combustion tubes were employed to allow a larger proportion of soda-lime for mixing with the substance.

2nd. In two cases (Nos. 3 and 4 of Table IV.) especial pains were taken to put the mixture of fine soda-lime and substance (about 1 part of substance to 40 of soda-lime) along the lower side of the large tube, and to cover it with fine soda-lime, so as to insure passage of the decomposition products through the covering layer of soda-lime, and thus secure more perfect contact of distillation products with soda-lime. The mixture was got into place by aid of a thin narrow tube inserted into the wide combustion tube.

How ineffectual the attempts were to secure complete ammonification of the nitrogen is shown by the figures of Table IV., in which the figures for "Nitrogen found" are those obtained by titration with standard alkali in the usual way. The acid in the bulb after combustion was turbid in every case.

TABLE IV.—*Percentages of Nitrogen obtained from Strychnine by Soda-lime.*

No.	Substance Used. Gram.	Internal Diameter of Tube. Cm.	Nitrogen found. Per Cent.
1	.5	1.25	6.47
2	.5	1.25	7.64
3	.2	2.	5.88
4	.2	2.	7.35
5	.1	2.	1.57
6	.1	2.	1.70
7	.1	2.	5.88
8	.1	2.	3.92
9	.05	1.25	1.96

In brief, the greatest care in pulverising the substance finely, mixing intimately with soda-lime, using excessive quantities of soda-lime, and packing the tube closely to avoid channel, were ineffective. Insuring the greatest contact of substance and distillation products with soda-lime and most careful heating evidently do not prevent escape of nitrogen in other forms than ammonia. Attempts to get all the nitrogen as ammonia by Kjeldahl's method were also unsuccessful.

The main outcome of the experiments above detailed may be summarised in few words.

1. In combustions with soda-lime, an open space (channel) in the tube is unnecessary and involves danger of loss of nitrogen. This loss may be due to nitrogenous distillation products which escape ammonification, or to dissociation of ammonia, or to both.

2. Prolonging the time of combustion unduly may induce loss. The most probable cause of this loss would seem to be dissociation of ammonia.

3. High heat increases danger of loss, probably by dissociation of ammonia, especially when open space in the tube or slowness of combustion, or both, leave the gases for a considerable time exposed to the action of the heat and not in intimate contact with the soda-lime.

4. With casein (and the data cited and to be cited in the other articles of this series imply that the same is true for the protein compounds of animal and vegetable tissues generally) sufficient contact between soda-lime and substance at moderately high heat

suffices to convert all the nitrogen into ammonia. This is effected by intimate mixture of soda-lime and substance, providing a moderately long anterior layer of soda-lime, close packing of the tube, heating the anterior layer well before the heat is applied to the mixture, and keeping it well heated until the operation is done.

5. There are, however, substances, such as strychnine, which, when heated with soda-lime, yield distillation products that are very difficult of ammonification.

A succeeding article will refer to these points in more detail, discuss loss by incomplete ammonification and dissociation, and refer to a probable explanation which they give of the failure of the soda-lime method to bring satisfactory results in some of the cases most frequently cited in evidence of its unreliability.

#### REVIEWS.

SOAPS AND CANDLES. Edited by James Cameron, F.I.C. London: J. and A. Churchill. THIS is the fifth volume of the series of technological handbooks issued by Messrs. Churchill. We have had occasion, when noticing the appearance of the former members of the series, to always speak in terms of commendation, and we are pleased to be able to do the same in the case of the book now under notice. Mr. Cameron has done his work well and has quite caught the style of matter required by the readers of this class of books. He has also had the advantage of the aid of such eminently practical men as Mr. Leopold Field and Messrs. Cook, whose information he suitably acknowledges in his preface, and the result is a handy little volume of 300 pages which is distinctly the practical handbook it professes to be. The illustrations and the basis of the whole headings are taken from the articles in "Cooley's Encyclopædia," but they are extended and brought down to date and placed in a form at once easy to read and inexpensive to buy. In issuing this series the publishers have followed the example already set by Messrs. Spon and other publishers of large encyclopædias, of cutting up the matter and republishing it in small books, and we fancy that the death knell is nearly rung of the ponderous tomes, which, starting with a flourish of trumpets, end by the first volumes being antiquated and practically useless before the last one is printed. It is much better that we should be provided with small and easily renewable books on particular subjects, and the work before us is a very good example of what such a book should be.

NATURE'S HYGIENE: A SYSTEMATIC MANUAL OF NATURAL HYGIENE, CONTAINING A DETAILED ACCOUNT OF THE CHEMISTRY AND HYGIENE OF EUCALYPTUS, PINE, AND CAMPHOR FORESTS, AND INDUSTRIES CONNECTED THEREWITH. By C. T. KINGZETT, F.I.C., F.C.S. Third edition. London: Baillière, Tindall, and Cox.

THIS work, originally written as a short book dealing with the author's investigations into the subject of peroxide of hydrogen and its development in the form of his disinfectant "Sanitas," has become gradually extended into a large volume of 439 pages. There can be no doubt that it is now a very interesting popular treatise on sanitary science from a chemical point of view, and that many of the author's ideas tend in the right direction. His theory of true disinfection is to regard the so-called disease germs as being best attacked by removing (by oxidation) the septum in which they grow. His views are best summarised by quoting from his preface, in which he says:—"I have throughout the book endeavoured, as opportunity offered, to clearly indicate the precise



relations, as cause and effect, which are now known to exist between micro-organic life and so-called infectious or contagious diseases. In this connection it is gratifying to be able to record the fact that, by the investigations of later years, it has been well established that micro-organisms do not produce disease in any direct manner or by their mere mechanical presence in or upon the body, but that the real poisons are of the nature of chemical (toxic) products which result from changes in the media in which the microbes develop and upon which they grow and multiply. This view I have for many years consistently advocated, although it received little credence until quite recently." Some of the chapters on burning sanitary questions, such as the oxidation of sewage in running streams, and the irrigation *versus* precipitation question, are well and moderately written, giving a terse, but fair, hearing to both sides, and summing the whole up in a sensible fashion. The later chapters contain an immense amount of experimental research on the action of disinfectants, and would doubtless have attracted much more notice among scientists had they not been of necessity conducted with a proprietary substance like *Sanitas*, but setting all such prejudice aside, it must be admitted that many of the results obtained are both striking and interesting. In conclusion we will give one other quotation, the views expressed in which will, we venture to think, receive endorsement by all our readers. "I renew the expression of my opinion, that chemical officers are urgently needed to act side by side with medical officers of health and engineers, to effectively deal with the many important questions affecting public health and the safety of the community at large, which were referred to in the preface to the second edition of this book. While the development of chemical science in recent years has been most marked, sanitary authorities have not sufficiently availed themselves of its services, but relied much too confidently on mere engineering skill. The disposal of London sewage, as at present effected, will serve to exemplify my meaning, and may be taken as by way of illustrating the disastrous results that may ensue from such unfortunate reliance."

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TREATISE ON ALCOHOL, WITH TABLES OF SPIRIT GRAVITIES. By THOMAS STEVENSON, M.D., Lond. Second edition. London: Gurney and Jackson.

ALTHOUGH now published under such a grandiloquent title, this is simply the second edition of the author's table-book showing the percentages of alcohol (by weight and volume) and of proof spirit, corresponding to the gravity of mixtures of alcohol and water. In arrangement these tables are practically the same as those previously published by Mr. Hehner in our columns, and laid before the Society of Public Analysts in March, 1880. Dr. Stevenson, however, claims a *raison d'être* for his tables by stating that Mr. Hehner's are wrong while his are accurate. The tables are prefixed by a few pages of matter giving a historical notice of the various researches that have been published on the specific gravity of alcohol, and an argument to prove that the experiments of Gilpin (whose data the author has adopted) are the only reliable ones. In the course of his argument the author says:—"We have no assurance of the accuracy of Fownes' table, and it appears to me to be inferior far in accuracy to the tables of Gladstone and Gilpin. Prof. R. S. McCulloch in an exhaustive report to the Secretary of the Treasury, U.S., in 1848, speaks, too, in disparaging terms of Fownes' work. Yet

Mr. Hehner, who has elaborated a set of tables of spirit-gravities, based on Fownes' tables, terms these "the excellent tables of Fownes," and adds "all later investigators have confirmed the general accuracy of Fownes' table." I have been unable to ascertain by whom the table referred to has been verified in detail; and I am assured on competent authority, that Mr. Hehner's tables are incorrect at some points to the extent of nearly one-half per cent. of proof spirit—a serious difference." We leave the battle of the tables to be fought out by their respective authors, and we will be curious to see what Mr. Hehner, who usually has the courage of his opinions, has to say to such a direct attack on his work. The preface to the present edition of Dr. Stevenson's book contains a delicious little bit giving us some idea of the labours that have to be undertaken by the author of a treatise who has to keep up with the advance of science. He says:—"In issuing a second edition of this book—originally published under the title of "Spirit Gravities"—I have felt it necessary to *re-write the whole work* (the italics are ours) in order to bring in an account of the recent investigations, chiefly of Messrs. Squibb, on absolute alcohol. It is not necessary to alter the tables, as these are sufficiently accurate for all practical purposes." The part thus indicated as the "whole work" extends to sixteen pages, while the tables occupy seventy-three pages. If it should really turn out that these tables are the most correct, then hundreds of analysts will buy the book, but it is very questionable if 10 per cent. of these will ever trouble to read the "whole work," the re-writing of which has apparently converted a good table-book into a treatise.

#### LAW NOTES. TO OUR READERS.

*Some years ago it was decided to discontinue the reporting of ordinary police-court proceedings, and only to give such cases as authoritatively established some point in connection with the working of the Acts in which public analysts are interested. After a fair trial of this system, a majority of the members of our Society have expressed a wish that our old practice should be, to some extent, returned to, and, in deference to such request, we have decided to resume the reporting of police proceedings. The line will be drawn at accounts of ordinary cases, and reports will only be inserted when any novel, legal, or chemical point arises, or where the certificate of the analyst is in any way attacked. Any member or subscriber connected with such a case is, therefore, invited to furnish us with a report of the proceedings, signed with his name, not for publication, but as a guarantee of exactitude.*

**ADULTERATION OF AMERICAN LARD—IMPORTANT PROSECUTIONS.**—At the Liverpool Police Court, on the 20th ult., before Mr. Raffles, several wholesale provision merchants in Liverpool were summoned for having sold lard not of the nature, substance, and quality demanded by the purchaser. Mr. Marks appeared to support the summonses on behalf of the Health Committee of the Corporation. The first case called on was one in which Cuffey Brothers, Victoria Street, were summoned, and for whom Mr. Pickford appeared. The court was crowded with representatives of the provision trade.

Mr. Marks, in opening the case, said the defendants, who carried on business at 40, Victoria Street, were summoned for selling lard which was not of the nature, substance, and quality demanded by the purchaser. The warehouse of the defendants was visited on the 14th May by Inspector Baker, an officer under the sale of food and drugs act. He there saw a number of buckets of lard, on which was printed, "N. K. Fairbank and Co., refined lard, Chicago." He inquired the price, and ultimately purchased a bucket for 10s. 8d. He gave the usual notice about requiring the purchase for analysis, and offered to divide it for that purpose. His offer was accepted. He then left defendants a sample, another portion he took to Dr. Campbell Brown in the usual course, and the third he retained. Dr. Brown furnished a certificate, upon which, as a rule, the case rested. Owing, however, to communications that had been made to him (Mr. Marks) by gentlemen instructed on behalf of the defendants, in this and other cases, it was thought desirable that Dr. Brown should be in attendance, so that he might give evidence in a more ample manner than would appear from his certificate, and in order that the defendants might have an opportunity of cross-examining him. The certificate which Dr. Brown had furnished stated that he had analysed the lard, and that in his opinion it contained considerably more than 40 per cent. of a mixture of cotton-seed oil and either mutton or beef fat. The court would probably gather this was a case of greater importance than cases under the sale of food and drugs act usually were. Certainly if the court was to be troubled with all the information which had been furnished to him officially, and also anonymously, he should imagine that so far as the United States were concerned, the people there were at present given up entirely to the lard question. (Laughter.) However, in the provision trade the case was undoubtedly regarded as of very great importance, and he was bound to say it was important to more classes of persons than one. It was of



vast importance to the makers of the lard, whose profits were simply enormous, and also to the consumers of the lard. It seemed that about eighteen months ago it was discovered that lard was being imported into this country which was adulterated. It was imported from America, and the fact appeared to have become known in America, and to have created a tremendous amount of feeling there.

Mr. Pickford objected to these observations as being irrelevant.

Mr. Marks said he was simply leading up to the facts. What he was going to say was, that the principal manufacturers of this lard were persons whose names appeared on the buckets, namely, Fairbank and Co., of Chicago, and Armour and Company. It was known to Dr. Campbell Brown about eighteen months ago that this importation of lard was going on.

Mr. Pickford again objected to Mr. Marks entering into matters not connected with the present case.

Mr. Marks contended that his observations had reference to the case before the court.

Mr. Raffles said he should rule that what Dr. Brown did eighteen months ago was not relevant, except it had reference to the summons now being heard.

Mr. Marks (continuing) said that in order to meet Mr. Pickford's objection, he would put the matter in this way—On examining the sample in question, Dr. Brown found that his researches, which had occupied him more than eighteen months, had furnished him with knowledge that enabled him to discover that the sample contained cotton-seed oil. All he was going to say before was that eighteen months ago Dr. Brown could not have done so. (Laughter.) During the last month when Dr. Brown analysed the sample, he found there was a considerable proportion of cotton-seed oil, and also of stearine, either mutton or beef fat. Lard adulterated in that way, Dr. Brown would show, was a very inferior article indeed, was not of the same value, and was not as useful for the purposes for which genuine lard was used. The cotton-seed oil produced in one season in the United States amounted, he believed, to 180 or 200 million pounds weight. Of that a very large percentage, pretty nearly half, was used by manufacturers of what was called refined lard, but which the prosecution suggested was adulterated lard. The price of cotton-seed oil was only about 22s. 6d. a cwt., and the price of beef fat only 30s. a cwt., whereas the price of pure lard was 42s. 6d. a cwt. Therefore it would be seen that when such large quantities of cotton-seed oil were used, enormous profits resulted to the manufacturer, who could substitute for the more valuable article a cheaper one. Every week there came into Liverpool from £20,000 to £30,000 worth of lard, and the whole of that he presumed was used in the preparation of food. It was consequently a serious matter for the consumer, as well as a matter of considerable importance to the fair traders in lard, who offered for sale the genuine article.

Inspector Baker deposed to visiting the defendants' warehouse, and purchasing a bucket of lard on which was the name of Fairbank and Co.

Mr. Pickford did not cross-examine.

Dr. Campbell Brown, examined by Mr. Marks, stated that on the 15th May he received the bucket of lard from the last witness and analysed it. The result of his analysis was that he found the lard to contain a very large quantity of cotton-seed oil and beef or mutton fat. He estimated the total quantity as considerably more than 40 per cent. He really believed the quantity was more than 50 per cent., but he was quite certain that it was more than 40 per cent.

Cross-examined by Mr. Pickford.—I suppose you mean the cotton-seed oil and the beef or mutton fat together made 40 per cent. ? Yes.

You don't distinguish the one from the other ? I have not done so.

Can you do so ? I can't tell the precise quantity of beef fat.

Then, I assume, if you can't determine the quantity of beef fat, that you can't determine the quantity of cotton-seed oil ? I am quite certain there was more than 30 per cent. of cotton-seed oil, but I can't determine more. I can't estimate exactly the quantity of beef fat, and therefore I can't tell precisely the quantity of cotton-seed oil over 30 per cent.

Does not your test tell you anything about the beef fat stearine ? It tells a good deal about it, but not the precise quantity.

How do your tests show the presence of these things ? I think you would require to attend a course of lectures on chemistry before you could do it. (Laughter.)

Mr. Raffles: I think we had better not have that.

Mr. Pickford: What kind of test do you use ? I put the whole thing through seven or eight processes and then argue the thing out. I have no individual test.

Supposing there was no cotton seed oil at all ? Yes, but there is. (Laughter.)

Supposing there was no cotton seed oil, but beef fat only added to the lard, could you distinguish that fat from the other fat ? I can distinguish beef fat from hog's fat.

These are, as a matter of fact, new tests ? They are new applications of old knowledge.

I meant that you could not until quite recently distinguish cotton-seed oil or beef fat in the lard ? I was quite certain about the cotton-seed oil fifteen months ago, but I did not see my way to get out the quantity sufficiently for judicial purposes. Four or five years ago I knew about a certain quantity of beef fat. I don't want to give my results now for two reasons. One is that I am getting the quantity less every week, and the other is I don't want to let the makers of lard know how little they can put in without detection. (Laughter.)

Mr. Pickford, for the defence, said his worship would probably have divined from the cross-examination that he was not going to deny the presence of cotton-seed oil in this refined lard. But he was going to say that "refined lard" was a perfectly well-known trade term, and everybody was

aware that it was a compound of fats and not real hog fat. If that was proved, and the effect of the notice on the barrel that it was refined lard meant what he said, then he should bring the case within the case which was decided about mustard, in which a man asked for mustard, and on the packet was a notice that the contents were not pure mustard but compound mustard. He did not think he was wrong in trying to stop what seemed to him to be irrelevant statements—of which a number had been made—about the comparative prices of the ingredients.

Mr. Marks: I can prove my statements if my friend wants.

Mr. Pickford: A great number of those statements are absolutely inaccurate.

Mr. Marks: I think it is only fair to myself to say what I based my statements upon.

Mr. Raffles: I have nothing to do with it.

Mr. Marks: I was basing my statements upon the general brokers' trade circular of the 15th of last month.

Mr. Raffles: Don't let us talk about it.

Mr. Pickford: No, sir; I don't want to. But if I allow it to pass without contradiction it would no doubt be stated in the papers that it was admitted as a fact, and I don't admit it. I do admit that this refined lard is sold at about the same price as pure lard, and I admit that as being important for showing that the people who use a very large quantity of this stuff in this country willingly pay the same price, and have no complaint to make of the stuff.

Mr. Pickford was proceeding to make remarks upon the summons when

Mr. Raffles said: The only question I have to deal with is the adulteration.

Mr. Pickford: But is it adulterated?

Mr. Raffles: On Dr. Campbell Brown's evidence it is adulterated.

Mr. Pickford: I say it is not adulterated. It is sold as refined lard, and I can call evidence—

Mr. Raffles: I can't go into that.

Mr. Pickford: If you say you can't hear evidence on that I can't say any more.

Mr. Raffles: I can't.

Mr. Pickford: But supposing that the meaning of the trade term "refined lard" is not pure hog's lard, then it is a compound and not adulterated.

Mr. Raffles: I can't go into any special meaning which is attached by the trade to refined lard.

Mr. Pickford: If the words "refined lard" do mean a compound of fats, it means that everybody who buys the stuff, that the purchaser is not getting an article of a different nature, quality, and substance to that demanded—

Mr. Raffles: Well, of course this case will go elsewhere, whatever my decision is.

Mr. Pickford: Then I understand that you reject any evidence on my point?

Mr. Raffles: Yes.

Mr. Pickford said he ought perhaps to state that he intended to show that refined lard was a well-known term, meaning a compound of fats and oil, and was not confined only to the American market, but the English refiners, he believed, use some cotton-seed oil. Refined lard, whether American or English, was never pure hog's fat, and everybody knew it.

Mr. Raffles: Dr. Campbell Brown tells me this is adulterated, and that is quite sufficient for me. You will take the case elsewhere, I suppose.

Mr. Pickford: It is very likely, but I can't say anything, one way or the other.

Mr. Raffles: It is a very serious question, and one which ought to be dealt with seriously. I shall inflict a fine of £5 and costs.

Messrs. Pelling, Stanley, and Co., Victoria Street, were summoned, and Mr. Mulholland appeared for the defence.

Mr. Raffles: What is the difference between this case and the last?

Mr. Marks: An important one in one respect, inasmuch as the lard is not of the same manufacture, but is from the other great refiner's. This also is called refined lard, but whether the defence mean to rely upon that or not I don't know.

Inspector Baker gave evidence of the purchase of the lard from the defendants, and stated that when he offered to leave a sample with them they declined to receive it. He then took the lard to Dr. Campbell Brown.

Dr. Campbell Brown said he analysed the lard in question, and found a very large quantity of cotton-seed oil and fat extracted from beef or mutton. He estimated the total quantity approximately at 40 per cent. The case was not so bad as the last.

Mr. Mulholland said that the witness had analysed two samples of lard of the same brand and had given different figures. He therefore applied for the case to be adjourned for the lard to be sent to Somerset House for analysis.

Mr. Raffles said he should decide the case himself.

In cross-examination, Dr. Campbell Brown said he believed that of late years the oil formerly taken out of hog's fat to make it harder and more fit for carriage was not now saleable, because of the introduction of mineral oils for lubrication.

Mr. Mulholland: And therefore it becomes commercially more important to put hardening stuff in the lard than to take out the oil?

Witness: It becomes important to the manufacturers, but it is not right.

Mr. Mulholland: You are not the analyst of right and wrong, but only of fat and stearine.  
(Laughter.)



Mr. Mulholland submitted that the case came within the first subsection of the Act, namely that these substances were not introduced for any fraudulent purpose, but for the purpose of hardening the lard and making it merchantable.

Mr. Raffles: It cannot be for the purpose of bringing it over. I am against you there.

Mr. Mulholland said he also took the same point as Mr. Pickford, and as his worship was against him there he supposed he would grant them a case on that as well as on other points if necessary.

Mr. Raffles: I will give you any means you may require for testing my decision. It is very desirable this matter should be settled. I shall inflict the same penalty of £5 and costs.

Messrs. J. and T. Edwards, provision dealers, Whitechapel, were summoned for a similar offence.

Mr. Edwards, a member of the firm, appeared and stated that he bought the lard as pure lard.

Mr. Marks: If you did I should sue the manufacturers for the amount of the fine and costs.

The invoice for the lard was handed to his worship, who, however, said it was no warranty of purity.

Mr. Edwards said he bought the lard as pure, and consequently he thought the fine ought to be less than in the other cases.

Mr. Raffles said he should inflict the same fine, £5 and costs.

**ALLEGED ADULTERATION OF WHISKY.**—Ellen Walker, the landlady of The Hotel, Tuxford, was summoned for selling whisky 30·8 under proof.

Frederick Wm. Holden, of Retford, inspector under the Food and Drugs Act, proved the purchase of a small portion of whisky, and produced the certificate of Dr. Otto Hehner, the county analyst, which stated the whisky was adulterated to the extent named, whereas the law permitted the strength to be 25 degrees under proof only. All this was admitted; but the defence set up was that a notice was posted up in the bar, the smoke-room, and elsewhere as follows:—"All spirits sold in this establishment are diluted, but not under half-proof strength," and Mr. Besoby quoted the case of *Gale v. Elsie* (L.R., Queen's Bench Division, vol. 10, p. 515) to prove that in the face of such a placard there could be no prejudice to the purchaser, and that, therefore, no offence had been committed. The presence of the notice was not denied, but upon the point whether it was so concealed by a larger poster as to be hidden from sight, much conflicting evidence was adduced. No decision was given on this contention.

A further point was, however, raised, namely, that, at the time of the purchase, the inspector must forthwith notify to the seller, "or his agent selling the liquor," that it was bought for analysis by the county analyst. Now the whisky was sold at a small window in the public-house by the barmaid; and although the inspector duly made the notification as required by the law, he made it not to the "person selling the liquor," but to the son of the defendant, who managed for his mother, and in a room close by, immediately after the purchase. It was upon this point that the charge was dismissed, but the court granted a case for appeal.

**"LARD CHEESE" PROSECUTION.**—Dennis M'Cashin, provision merchant, 765, Gallowgate, appeared before Sheriff Balfour, in answer to a summons charging him with having, on 10th May, sold a pound of cheese to Robert Inglis, sanitary inspector, the fat of which consisted essentially of isomargarine or foreign fat. The respondent said that he bought the cheese as Danish cheese, and was not aware that there was anything wrong with it. He was told by the man who sold it to him that it was good, and not having an experience in the trade he believed this. Had he known there was a risk in selling it he should never have purchased it. In answer to the Sheriff, M'Cashin admitted that on the invoice which he received from the dealer the cheese was termed "lard cheese." The Sheriff said that in terming this lard cheese the seller had protected himself, but the accused had not, because it had been held that a simple invoice was not a guarantee. The accused required to be protected by a guarantee. He evidently knew this was lard cheese before it was sold, and he ought to have told the purchaser that it was such before selling it. There was no harm in selling lard cheese if the purchaser knew what he or she was getting, but it would not do for a person to be served with it who went into the shop to get milk cheese. His lordship imposed a penalty of £3.

**SELLING MARGARINE FOR SALT BUTTER.**—Before Sheriff Dove Wilson, at Aberdeen, William Allan, grocer, Gallowgate, was charged with having on the 26th April, sold and delivered to James Mackintosh, sanitary officer, an article of food—namely, a quantity of fatty matter which was not of the nature, substance, or quality of the article demanded—namely, salt butter, in respect that it contained fat or foreign matter to the extent of 87 per cent. He pleaded not guilty. Alexander Davidson, the grocer's assistant, in answer to the Prosecutor, said that he told Mackintosh at the time he had no salt butter, but that he had margarine. He told him it was margarine because he was informed of the purpose for which it had been bought. They were in the habit of supplying customers who asked for salt butter with margarine without any remark. They had no salt butter in the shop on the day in question. Further evidence having been given, the charge was found proven, and in giving judgment the Sheriff said it did not seem to his Lordship that to sell a quantity of margarine as butter was protected under the Act simply because at the time of the sale the seller wrapped the article in a wrapper upon which the word "Margarine" was printed. The section of the Act applied evidently to cases where there was a more or less accidental or necessary mixture, and was never meant to apply to a case like the present. It was never meant that the public, after being supplied with one article should have



no remedy because the name of another article was marked upon the parcel. As the case had evidently been brought to settle the question whether this practice was right or wrong it was not necessary to impose a heavy penalty. His Lordship took the opportunity of saying that so far as his knowledge went margarine was a perfectly inoffensive substance. There was no reason whatever why it should not be sold, provided always it was sold openly, and nobody sold the article either deliberately or tacitly representing it as an article of a higher quality. That appeared to have been done here, and therefore the penalty would be one of £2.

### CORRESPONDENCE.

*[The Editor is not in any way responsible for opinions expressed by his correspondents.]*  
*To the Editor of the ANALYST.*

SIR,—In his article "On Adulterated Lard," in the April ANALYST, pp. 69 and 70, Mr. Stephen P. Sharples, State Assayer of Massachusetts, makes some remarks that will bear commenting upon.

In the first place he states; "As is well known cotton-seed oil is a semi-drying oil, having strong siccative properties at the temperature of 212° F., this admixture unfits the lard for many uses. It is impossible to make good biscuits with such a compound, as they rapidly become rancid."

We presume his term "biscuit" is equivalent to "cracker." The amount of lard used in cracker-baking is almost infinitesimal, compared with the lard used for other purposes. Cracker-bakers never use anything but hog lard, and the manufacturers of refined lard do not attempt to sell their product to cracker-makers, though for all other purposes it is as good if not superior to the hog product.

In order to test the charges made against cotton-seed oil by cracker-makers, the writer had a batch of crackers made, in which a mixture of cotton-seed oil and beef stearin was substituted for lard. The crackers when first made had a better flavour than if lard had been used, and it was four months before they showed any indication of the rapid rancidity mentioned by Mr. Sharples. At the end of six months they were rancid, but not enough to prevent their being eaten.

Some jumbles baked eight months ago with the same compound are as sweet as the day they were made.

Second: "The refining of lard consists solely in adulterating it with cotton-seed oil and oleo-stearin."

Mr. Sharples has never refined any lard, or he would not make such a statement. To begin with, the lard has to be carefully settled to remove the suspended particles of animal tissue, and the small percentage of tank water left in by the rendering. The water and membranous matter will range in prime steam lard from 0.25 to 0.50 per cent., and is bad for the keeping qualities of the lard. Often settling alone is insufficient, and the lard has had a washing with a little weak lye to remove the impurities.

The oleo-stearin has to be carefully selected and refined, and the same is true of the cotton-seed oil. English cotton-seed oil made from Egyptian seed would not be suitable for refined lard, and in America would be used for soap or lubricants.

Mr. Sharples neglected to state that lard refiners press a large amount of their lard in order to obtain "lard stearin," which is used in the refined lard to give it the requisite hardness. Since the inauguration of the oleo-margarine industry the packers have systematically robbed their lard of the "leaf," or omentum fat, and have thus reduced the melting-point of commercial lard. As lard of two melting-point has little value in making light pastry, the lard refiner has to overcome this difficulty by the addition of lard stearin, or oleo-stearin in proper proportions to make the lard give the best results.

Regarding the Belfield test, I would say that while at times it gives very characteristic crystals, at other times the crystals are not definite enough to be relied on implicitly. The nitric acid test is apt to be uncertain, as old samples of lard will show a marked coloration in the absence of cotton-seed oil. Same is true about the sulphuric acid test.

The Bechi test is at times uncertain, owing to the method of refining used on the cotton-seed oil. On old samples of oil it sometimes gives negative results, while with pure lard oil it will sometimes give a slight reaction, especially old samples.

Chicago, U.S.A., May 15th, 1888.

DAVID WESSON.

*To the Editor of the ANALYST.*

SIR,—My attention has been called by Mr. Allen, and also by Mr. Jones, of Wolverhampton, to an error in my paper on "The iodine absorptions, combining weights, and melting points of certain fatty acids," which appears in the ANALYST, May 1888, p. 89. In the first column of the table of results, "Name of oil or fat" should read, "Name of fatty acids."

As this mistake may possibly lead to confusion, if not corrected, I shall be obliged if you can publish this letter in the next number of the ANALYST.—I am, etc.,

Manchester, June 22nd, 1888.

ROWLAND WILLIAMS.



# THE ANALYST.

AUGUST, 1888.

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### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS. POLLUTED DRINKING WATER AND THE CLOSURE OF WELLS.

BY ALFRED HILL, M.D., F.I.C.

(*Read at the Meeting, June 20, 1888.*)

PROFESSIONAL and even public opinion has long been opposed to the use, for drinking, of water polluted by excrementitious matters or sewage; the use of such water not only is repugnant to sentiment, but experience has proved that it sometimes produces serious disease and death. This fact has been recognised from the earliest historic times, and in the more enlightened and more scientific modern times it is still more frequently brought home to us. For the last half-century the subject has received greater attention than ever before, till it has come to be generally acknowledged that sewage-polluted water cannot be drunk with safety. This widespread impression has led, in many large communities, to the obtaining of Parliamentary powers to supply pure water from a distance, either from the surface of the earth in districts free from population, as Glasgow from Loch Katrine, Manchester from Thirlmere, and Liverpool from the source of the Severn, as Rome did from the distant Arno; or from the strata of the earth at points so deep as to be beyond the influence of surface filth, and to the abolition of the surface wells of such towns at one sweep.

These special and local Acts, as well as the Public Health Act, have served not only to confirm public opinion in favour of pure water, but they have also equally led the public to believe that it was only necessary to prove, either by common observation or by chemical means, the pollution of a water supply with animal matter, in order—in cases of wells especially—to enable the closure of such supplies to be effected. In some towns there has, however, been no general closure, but each well is chemically examined in detail, and closed if the circumstances warrant this proceeding. On the mere proof of such pollution hundreds of thousands of wells in the country have been closed: in some cases—notably with the Broad Street pump at Westminster—after injury being proved, but in most cases without any such proof whatever.

Two cases recently decided, however, have excited much surprise and disappointment, inasmuch as orders to close the polluted wells were refused, on the ground that it had not been *proved* that the waters were “injurious to health”; these words are from the 70th Section of the Public Health Act, 1875. One of these cases occurred at

Enfield; the other at Birmingham. The Enfield case was brought before the Society of Medical Officers of Health on the 18th of May by the analyst, Mr. Lloyd, who raised the question, What, in the words of the 70th Section of the Public Health Act, constituted "water injurious to health?" He said the presence of sewage matters in potable water, even when in large quantity, appeared not necessarily to do so, though if the sewage contained the germs of disease it would; and he mentioned eminent authorities who had shown "that no sufficient chemical variation, to be of a decisive character, existed," to indicate when pollution is injurious and when not. Bacteriology, he said, was equally at fault in proving the existence of disease germs. Recognising these facts, he had, he said, when reporting upon contaminated water, simply stated the water contained sewage, and was therefore unfit for drinking purposes, *and this had sufficed in most cases to ensure the closing of the wells*; but recently a contested case at Enfield was lost on the ground that the report did not use the words of the Act. The water in question contained:—Total solids, 83·72; loss on heat, 7·0. Oxygen absorbed, 0·157; Cl, 8·40; N, as nitrates, 2·28;  $\text{NH}_3$ , ·084;  $\text{Alb}^d$ .  $\text{NH}_3$ , ·008. No water analyst of experience could, it would be supposed, do otherwise than condemn such a water, yet, singularly enough, two other chemists, whose analytical results agreed with Mr. Lloyd's, and who made their analyses independently, gave favourable reports of the water.

"The owner of the well disputed his report, and an analysis on his behalf was made by Mr. Gregory, who found practically the same result, with the exception of the nitrates, which he gives at 4·22, and who reported 'a perfectly safe water to use for drinking and domestic purposes, being free from any trace of sewage contamination.' In view of the conflicting evidence, the magistrate decided to send a sample to Dr. Bell at Somerset House, and he, in his report, stated that his results did not differ materially from the previous analyses. It was evident, he thought, from the large quantity of saline matters present, that it passed through a stratum of earth largely charged with sewage or organic refuse, and carried down with it various products resulting from the oxidation of the organic matter, which, though not dangerous to health, was very objectionable. As to how such a sentence came in the Act, he thought it was taken for granted that the mere presence of sewage in potable water was *prima facie* evidence that the water was injurious, and that view was supported by the fact that in the Sixth Report of the Rivers Commission on Domestic Water Supply river water receiving sewage, and shallow well waters were designated dangerous, without any consideration of the quantity of pollution. So long as Somerset House was made the court of appeal, it practically amounted to an inability to close polluted wells."

I have stated this case at some length because it is almost on a perfect parallel with the Birmingham case, in which the water was very similar, being better in some respects, but worse in others. The analytical results obtained by myself in 100,000 parts were as follows:—Total solids 148·2;  $\text{NH}_3$ , ·001;  $\text{Alb}^d$ .  $\text{NH}_3$ , ·015; N as nitrates 6·6; chlorine 11·2.

As in the Enfield water, however, the point on which the whole case turned was the word "injurious." In my evidence I said the water presented all-round evidence of pollution with matter derived from sewage; it was therefore dangerous for domestic use. The counsel for the defence asked me this question: "Seeing before you the defendant, who is now in perfect health, and who has drunk the water daily for twenty-five years without any illness, do you mean to swear that, if he were to drink now a pint, or a quart of the water, or more, it would prove injurious to his health?" I replied, "No; it might not at the present time, though it might; or, if not now, at some future time." The magistrate then asked me, "Do you say this well is injurious to health?" to which I replied, "No; I say it is dangerous, and likely to injure health at any moment." The



magistrate then said, "I think we must dismiss the case." Mr. Parker, the Inspector of Nuisances: "On what ground, your worship?" Mr. Goodman: "You have not proved that the well is so polluted as to be injurious to health." It was true I had not, because I could not point to illness or death which had been produced by it, and I know of no other proof; but I had proved all that chemical analysis enabled me to do—viz., that it was polluted in the same manner (that is, by animal matter) as many wells are which have been known to produce illness and death. Surely it was never intended by the Act that polluted wells are not to be closed until disease or death have resulted from their use; and yet, as I had proved everything short of this, I don't know what other proof was left to be produced.

The failure of the Enfield and Birmingham prosecutions is not only disappointing, but very embarrassing in its influence on future action. I don't know how Mr. Lloyd has fared with his Authority, but in my case all the blame is put upon my shoulders, because I did not swear that the water was "injurious," and so comply with the wording of the Act. But how, as a conscientious witness, could I do this when I know, as we all know, that water contaminated by normal or uninfected sewage is drunk every day by millions of people with apparent impunity? My answer to the categorical question put to me by counsel as to whether a draught of the Birmingham well water in question would now injure the defendant would at once have neutralised any opinion of mine to the contrary in the minds of the Bench, and so the sacrifice of my convictions would have done no good, while it would have left me discredited in the opinion of conscientious men competent to judge of the question, and an object of life-long contempt to myself. A scientific witness is not an advocate; he should not be a partisan. It is his bounden duty to tell the truth, as far as he believes he knows it, "without fear, favour, or affection," and regardless of all consequences; he is not responsible for them; and when in any case, to whatever extent he may unjustly be made to suffer in "mind, body, or estate," he cannot be brought by any power to a lower condition than that of the defeated general, who, in retreating, cried, "All is lost but honour."

Less with the object of obtaining confirmation of my views than to educate and satisfy the doubts of others, I wrote to several acknowledged eminent water analysts and medical officers of health, asking their opinions on the words "injurious" and "dangerous." The following are some of their replies, the first three being from gentlemen having occupied and occupying the most important chemical and medico-chemical positions under Government—viz., Dr. Frankland, of the Rivers Pollution Commission; Dr. Dupré, Analyst to the Local Government Board; and Dr. Thomas Stevenson, Official Analyst to the Home Office:—

[Copy.]

"The Yews," Reigate,  
May 26th, 1888.

MY DEAR DR. HILL,

In reply to your letter of the 19th inst., I am decidedly of opinion that no analytical results can justify the opinion that a particular sample of water is *injurious* to health, unless actual diseases are detected; but if the sample contains palpable evidence of pollution by animal organic matter, it is undoubtedly *dangerous* to health, inasmuch as it might be actually injurious at the time the sample was taken, or may become so at any moment.

With regard to the personal execution of analyses, it is neither possible nor desirable that the principal of a laboratory should personally make the analytical determinations; but they should be made under his direction or supervision, and the evidence of the assistant who actually makes them should be tendered if the results are challenged. I have often known this done, but I do not remember a case in which the assistant was actually called.

Believe me, yours very truly,  
(Signed) E. FRANKLAND.

[Copy.]

Laboratory, Westminster Hospital Medical School, Caxton Street, Westminster, S.W.,

DEAR DR. HILL,

June 15th, 1888.

There cannot be the slightest doubt that the well water, details of the analysis of which you have furnished to me, is polluted to a very serious degree by sewage or similar matter—in fact, it is little else than sewage oxidized to a greater or lesser degree by filtration through soil. Such a water is totally unfit for use. At the same time, I quite agree with you that you could not conscientiously have sworn that the particular sample of water you analysed was *injurious to health*. All that a chemist can do in such a case, seeing that we have as yet no means of identifying any of the germs producing typhoid fever, cholera, etc., etc., is to state that the water is polluted by animal matter—probably excrementitious—and that it *may at any time* become injurious. What I usually do in such a case as this is to condemn the well, rather than the particular sample of water. If a water shows sewage pollution it may or may not be injurious; that depends upon circumstances at present beyond the ken of the water analyst, but at all events it proves that the well from which the sample of water has been taken must be in more or less direct communication with sewage matter, and, whenever such sewage matter contains the germs of disease, the water from the well will be dangerous to every one drinking it.

Yours very truly, (Signed) A. DUPRÉ.

Chemical Laboratory, Guy's Hospital, London, S.E.,

DEAR DR. HILL,

June 16th, 1888.

I have read with much interest your case of the refusal of the magistrates to order the closing of a well where you testified that the water was *dangerous* to health, although you very properly declined to say that the water was actually injurious to health; and I have the figures of your analysis before me.

I think you were quite right in your opinion, and also in using the expression "*dangerous*." I am in the habit of using the term "*dangerous*" in my reports on such waters as this one you analysed, and of purpose. They are waters which expose the drinkers to risk, although individuals may again and again drink them without actual resulting harm. Without it can be shown that "*injurious*" and "*dangerous*" are synonymous terms, "*dangerous*" is the better and more correct expression.

Should the decision in your case be upheld, in future it will be well to use the word "*injurious*" with the qualification—*i.e.*, "*likely to injure health*"—or to have the law altered.

Believe me, yours very faithfully, (Signed) THOS. STEVENSON.

From H. Tomkins, Esq., M.D., B.Sc., Medical Officer of Health, Leicester.

Borough of Leicester, Health Department, Town Hall,

DEAR DR. HILL,

16th June, 1888.

*Re "Polluted Water."*

The analysis of the water (of which you sent me a copy) is sufficient to warrant its being condemned without hesitation, and I cannot understand your magistrates refusing to do so in the face of your evidence.

I have constantly condemned water less polluted than this, and have had no difficulty in getting wells closed on certifying that such is dangerous to health. Of course the absolute proof of its injury could only be sworn to *after* illness had been caused by it; but it appears to be a very anomalous position for any authority to be in, whose duty is to *guard the public health* against danger, to wait until injury has actually resulted.

I agree with you that, if justices take this view of the wording of the 70th Section, the sooner it is amended the better.

Yours faithfully, (Signed) H. TOMKINS.

From Matthew A. Adams, Esq., F.R.C.S., Medical Officer of Health, County Analyst and Analyst for the Borough of Maidstone.

Ashford Road, Maidstone,

DEAR DR. HILL,

16th June, 1888.

I am quite of your opinion as to the danger of allowing such a water as you send me particulars of to be used for drinking purposes. The great excess of solid matter (unless otherwise explained), the high chlorine, and particularly the great excess of nitrates, make it, in my opinion, imperative that the well should be closed because of the constant risk to those drinking it.

Of course it is impossible for anyone—chemist or other—without actual demonstration to say that any polluted water, for the time being, is positively "*injurious*," and it is unreasonable to



suppose that the P.H. Act would require an *analytical opinion* on the subject, if the injurious properties of a water could be otherwise, legally and more directly, demonstrated. It would hardly do to administer the water in question to a *susceptible person* to test the opinion. The fact is, as all properly informed persons well know, an opinion in such cases can be based on inference alone.

Perhaps the enclosed "*extract*," which further explains my views and my mode of dealing with such cases, will interest you.

Yours faithfully,

(Signed) MATTHEW A. ADAMS.

From Dr. Walter J. Sykes, late Medical Officer of Health and Public Analyst for Portsmouth.

The Laboratory,  
11, Billiter Square,  
London, E.C.,

DEAR DR. HILL,

June 18th, 1888.

I duly received yours enclosing analysis of a sample of water. There can be no two opinions on the matter; it is an exceedingly polluted water, and totally unfit for human consumption. I consider your qualification "dangerous" to be the correct one; it is a water receiving a large quantity of animal organic pollution, and, though at present the large amount of this is being oxidised into harmless nitrogen compounds, yet no one can foresee how soon this action may cease; and when it does, either temporarily or permanently, disease and probably death will be the result.

I have had to regret on many occasions that the bench adopt such literal interpretations of the wording of the Public Health Act. It has several times happened to me to be unable to get "dangerous" drainage arrangements altered, because I could not positively say they were "injurious" at the time. This is a great stumbling block in the way of preventive medicine, although we are able to point out dangers ahead, we cannot get them removed, but we must first wait until some serious illness or death has been the result.

Believe me, yours faithfully,

(Signed) WALTER J. SYKES, M.D., D.P.H.

From Dr. Alfred Ashby, Medical Officer of Health and Public Analyst for Reading Urban Sanitary Authority.

Town Hall, Reading,

June 18th, 1888.

DEAR DR. HILL,

There can be no doubt the water you allude to in your letter ought not to be allowed to be drunk; but, although I have frequently got polluted wells closed on similar evidence to yours, have long anticipated that such a line of defence as that you describe would be taken in some polluted water case, owing to the unfortunate wording of Section 70 Public Health Act, 1875.

Of course we all know that polluted water may be drunk for a long time without causing actual disease, in the same way that any unhealthy condition within the meaning of the 91st section of the Public Health Act may exist for a long time without actually causing disease; but at the water is under the most favourable conditions for widely disseminating disease as soon as the infective material is added to its ordinarily contained filth.

If these sections are to be interpreted in their literal sense, and we have in all cases to prove that some actual person has been made ill before proceedings can be successful, the sooner we give our work the better. Such an interpretation is certainly opposed to the spirit of the Act of 1875, which is preventive; and, if we were always bound by it, the greater part of the improvement in the public health which has been effected during the past fifteen years would not have taken place.

The evidence you gave in the case you refer to was perfectly correct, for you could have given no other reply to the question put to you.

I am, yours very faithfully,

(Signed) ALFRED ASHBY.

I have been asked if I could not see my way to saying that I regarded the words "injurious" and "dangerous" as synonymous. Unfortunately this is impossible, because I have, like Dr. Frankland, Dr. Dupré, and others, already and always rigidly maintained the distinction. As long as the single hair is strong enough to support the sword under the head of Damocles, who sits beneath it, his life is only in danger; if that hair should break he will be injured. The thoughtless person, smoking his pipe while sitting in a barrel of gunpowder, is only in danger as long as the spark and the powder are

kept apart; but in the opposite case he will be certainly injured. Surely the distinction between danger and injury is plain enough, but is it wise to wait for the breaking of the hair or the explosion of the powder before taking steps to prevent injury? That the object of the Public Health Act is to *prevent* injury is undeniable, and I maintain, therefore, that the sanitarian having pointed out the danger, it is for the legal tribunal to read and interpret the Act in that sense—I mean as a preventive Act—and order the removal of the danger. In the *Sanitary Record* of last month is a report of two wells which were closed at Halifax. The analysts, Mr. Ackroyd, of Halifax, and Mr. Jarman, of Huddersfield, reported in accordance with my view of the case. The former said that the waters were “contaminated and *dangerous* to drink”; the latter, that they were “polluted with sewage or drainage from manured land.” I suppose that if there had been no evidence beyond this the case would have broken down, but I hear indirectly that Mr. Ainley, the Medical Officer of Health, in agreeing with the chemical reports, went further, and swore the waters were injurious to health, although there was no actual proof of it.

Probably if the Birmingham case had had the benefit of a legal representative to conduct it, such a view would have been put forward and supported by the context of the section as would have influenced the court to regard the case differently; but there was little chance of my obtaining a different decision, altogether unsupported as I was by legal or other aid, while opposed by two solicitors, two eminent and leading counsel, and, as I am informed, six chemists, headed, I know, by Dr. Attfield and Mr. Wanklyn. Such an array of legal and chemical machinery, with a bench not qualified by special training, either legal, medical, or chemical, to adjudicate on such a question, was, of course, entirely irresistible.

A refusal of a closing order was almost inevitable, but even in that case all was not necessarily lost, for our legal advocate, if we had had one, could still have asked for a case on which to found an appeal, and I have little doubt that a higher court would have ruled otherwise.

Now, it appears to me that the question for this Society to consider is what is to be done under the circumstances, and I shall be very glad to hear the views and obtain the decision of my fellow-members upon it. It appears to me there are three ways of overcoming the difficulty, for of course we are all anxious to find a way out of it in the interests of public health.

The first, about which, however, I am not very sanguine, is to get magistrates to accept the secondary meaning of injurious—that is, “tending to injure,” “calculated to injure.” If this signification of the word were taken, we could all use it without hesitation, and so comply not only with the spirit, but with what is much more appreciated generally—with the absolute letter of the Act.

Secondly, a case properly conducted by a legal representative might be taken into court on the evidence that the water was “dangerous and unfit for use,” and, on an order for closure being refused, a case asked for, when, on appeal, the question would be argued to a satisfactory termination.

Thirdly, with or without such test case and appeal to a higher tribunal, the Government could be memorialised by Local Authorities, medical and chemical societies, and others to amend the 70th Clause so far as to bring it into conformity with the practical common-sense and science of the day and the reasonable demands of sanitary progress.

#### DISCUSSION.

The PRESIDENT said that the subject which Dr. Hill had brought before the Society was one of extreme importance to everyone. It seemed to him almost im-



possible for anyone to affirm positively that a polluted water would necessarily be injurious to health, remembering that such active poisons as opium and arsenic were habitually taken by individuals without injury to health, and that habit enabled the doses to be enormously increased. It would not be possible to say of a water that actually contained arsenic, that it would be necessarily injurious to the health of drinkers, although certainly it would be dangerous to health. In Sheffield they had recently some hundreds of cases of poisoning by lead, but the instances in which people had suffered were only a fraction of those in which people had drunk the same water with impunity, from which it must be concluded that only those people who were unusually susceptible to the influence of lead were affected under the conditions which prevailed. Similarly with sewage-contaminated water, which might be drunk by many persons, and especially those habituated to it, with impunity, while others would be immediately rendered dangerously ill. Of course too, as had been remarked, much depended on whether the sewage was excreted by persons in health, or those suffering from disease. The only possible way of ascertaining that a particular water would be actually injurious would be to experiment on human beings who had been previously accustomed to drink water of unexceptionable origin. A single experiment of this sort even would not suffice, as personal idiosyncrasy might prevent the poison from acting. He mentioned this to show how impracticable it was to affirm positively that a water was injurious to health. In the case of water which had suffered contamination by sewage, and in which the polluting matter had subsequently undergone oxidation, as was the case with the sample in question, he was in the habit of certifying that a change of conditions might render the water positively dangerous.

It was a great misfortune that, owing to circumstances well known to the members of the Society, many medical officers of health, and some professional chemists, were in the habit of ignoring the evidence of previous pollution furnished by the presence of nitrates and nitrites, and this in the face of the River Pollution Commissioners' results, which showed that filtration of fresh sewage through five or six feet of gravelly soil caused the oxidation of all the ammonia to the so-called "harmless" nitrates and nitrites. But if the process of spontaneous purification by oxidation were interrupted, as by the pores of the soil becoming saturated with water through heavy rains, or other causes, the oxidation was liable to be incomplete, and the character of the water would suffer a corresponding change.

With regard to the particular case of Dr. Hill, it was lamentable to think that any professional chemist should have been ready to appear for the defence of such a case. He could not quite understand how it was that there was no lawyer to conduct the case. Did Dr. Hill suggest that he could not have had the service of a lawyer if he desired? It seemed to him that the case was just one of those where the recognised solicitor of the Society might have been advantageously retained.

Mr. LLOYD proposed that the Society should act in conjunction with the Society of Medical Officers of Health in bringing the question before the Local Government Board, with a view to getting the objectionable clause altered. He protested against the tendency of magistrates to regard Somerset House as a court of appeal, and deplored the effect that Dr. Hill's case would have on the future decisions of magistrates, who would take it as a precedent, making it difficult to close any well. It was well known that a polluted water might be drunk habitually with impunity by persons long accustomed to it—probably having become so by degrees as its impurity increased—while a stranger coming into the district might experience very serious effects. He called attention to the terms of Dr. Bell's report on the water—namely, that it appeared to have filtered through a stratum charged with sewage or other decomposing animal matter, whereby

it was rendered objectionable, though not absolutely dangerous to health. Some outbreaks of diphtheria have occurred in Halifax which are probably to be ascribed to the use of water from shallow wells contaminated with sewage. He concluded by emphasizing the necessity of the case being taken up by the Society of Public Analysts.

Dr. SEATON, speaking for the Council of the Society of Medical Officers of Health, agreed that the matter must be thoroughly looked into. At Nottingham and Birmingham many wells had been closed without trouble or dispute. Nuisances—*e.g.*, those arising from the use of privy-middens—which are analogous to this, were satisfactorily dealt with at Nottingham, until the same point was raised, and with the same result—*i.e.*, interruption of the work of alteration; but at Bradford the Corporation came to the opposite conclusion on the same question. It was necessary, therefore, that a definite legal decision should be obtained.

Prof. BISCHOF, though at first misunderstanding Dr. Hill's position with regard to the use of the word "dangerous" instead of "injurious," on grasping his meaning, fully endorsed his views. He would suggest that an attempt should be made on the next occasion to induce the magistrate to accept the phrase, "likely to be injurious to health," as complying with the requirements of the Act.

Mr. ADAMS related a similar case which had happened to himself. A well contained a large quantity of nitrates, and, as he could not swear that it was literally "injurious to health," the magistrates refused to close it. In consequence, the Local Government Board, who had instituted the case, announced it as their opinion that it should have been referred to a "more capable man," who would have been able to make a statement sufficiently positive to cause the magistrates to close the well.

Dr. DUPRÉ said this was not the first case of the kind. Mr. Hehner and himself were engaged in a similar case some years ago in the Isle of Wight, when the magistrate, after hearing the case, said, "Now, sir, the case is dismissed, but if any one of your customers is seized with cholera you will be held responsible," and yet the magistrate did not feel justified in closing the well. He should like also to mention that some years ago he made a great number of analyses for Dr. Buchanan of waters purposely polluted. To one of these Dr. Cory had added a minute quantity of typhoid stool, and neither Dr. Frankland nor himself discovered the pollution, and this is what Dr. Buchanan wrote, chiefly in consequence of that analysis and of the outbreak which happened at Caterham, where a man suffering from typhoid fever had misconducted himself while down a well under repair:—"The chemist can, in brief, tell us of impurity and hazard, but not of purity and safety. For information about these we must go, with the aid of what the chemist has been able to teach us, in search of the surrounding water sources and affecting water services."\*

Now it should be borne in mind that this was written, not in relation to a water obviously polluted, but mainly in regard to waters so slightly polluted that the chemist was unable to find it out. Dr. Buchanan is, therefore, strongly of opinion that whenever analysis shows sewage pollution the water might at any time become injurious to health. In his (the speaker's) reports, whenever he found evidence of sewage contamination he always emphasized the fact that the well being within the influence of sewage might at any time become injurious to health. So far as he knew there were no means at present in the hands of the chemist to enable him to say that a water was polluted by any definite disease germs, and, therefore, whenever he could prove sewage pollution he should condemn the well, not because the water was necessarily injurious at that time, but because, in certain events, *i.e.*, the occurrence of an infectious disease among the people contributing to the pollution of the well, it would become at once highly injurious. It should

\* Report of Medical Officer for 1881, p. 21.



to be borne in mind that an impure water might be drunk for years by people who have been accustomed to it, but strangers coming into the neighbourhood might suffer immediately.

Mr. HEHNER urged the alteration of the word "injurious" to "dangerous," and moved a resolution to the effect that the change should be attempted to be made.

Dr. HULSE asked Dr. Hill what was the attitude taken by the chemists who were on the side of the defence.

Mr. CASSAL was of opinion that the phrase "injurious to health" was sufficiently elastic for the analyst to swear that such a water as that under discussion answered to that description. Mr. Fox has said that no person habitually drinking diluted sewage ever a picture of health. The question as to the "injurious" character of the water was eminently one for a medical man. What would he say under the circumstances? He asked Dr. Hill whether or no the water sediment was examined microscopically.

Dr. HILL, in reply to Mr. Lloyd and Dr. Dupré, said that he also knew of a case in which a stranger had suffered from drinking water innocuous to the inhabitants of a certain locality. He could not agree with Mr. Cassal as to the elasticity of the phrase "injurious to health," and thought that the position of a witness adopting that view would be untenable when exposed to cross-examination. He had not examined the deposit from the water, because it was only slightly turbid. With regard to what the President had said about the lead in Sheffield water, he did not think the cases were parallel. Lead is a tangible, easily detectable poison, but that present in impure water is not. He agreed that the nitrates should not be ignored; one could judge of a water, not of a man, by the company it kept. In respect of the question why he had not obtained legal advice, he had suggested the desirability of having a solicitor or barrister to conduct the case to the Inspector of Nuisances, who, however, thought it unnecessary. The case was decided summarily, and the chance of appeal lost from want of legal advice.

At the conclusion of the discussion on Dr. Hill's paper, Mr. Otto Hehner proposed, Mr. Bostock Hill seconded, and it was unanimously resolved,

"That a deputation from the Society of Medical Officers of Health and the Society of Public Analysts wait upon the President of the Local Government Board, and urge upon him the necessity of altering the wording of the 70th section, 'injurious to health' to 'dangerous to health.'"

#### FURTHER NOTE ON PEPPER ANALYSIS.

By CHARLES HEISCH, F.I.C.

(Read at Meeting, June, 1888.)

WHEN some time ago I laid before you the results of some analyses of pepper, I considered that the relative amounts of starch formed a fair basis for calculating the amount of any adulterants. It has been objected that the boiling with acid, and subsequent determination of rotating power, gave uncertain results. After carefully repeating my experiments, I have come to the conclusion that if always boiled for the same time, the results do not differ materially from the truth when we work on pure starch. Having repeated the pepper experiments, not only by boiling, as before, for three hours, but continuing the boiling for four hours without materially affecting the results, I still hold that much may be learned in this way. As, however, poivrette and some other adulterants consist in great part of cellulose, I thought it possible that the amount of solid body might be taken as an index of the amount of adulteration with such matters as poivrette. I therefore determined the amounts of cellulose in many of the

peppers before analysed, but I found such wide variations in the amount in undoubtedly pure peppers, that I could not draw any very reliable deductions from it.

PERCENTAGE OF CELLULOSE IN ASH AND WATER FREE PEPPERS.

1. Ashen Penang,	black .. .. .	15.08
2. Trang,	" .. .. .	11.58
3. Singapore,	" .. .. .	14.61
4. " another sample,	" .. .. .	14.32
5. Tellichery,	" .. .. .	12.92
6. Penang,	" .. .. .	27.82
7. Light Dusty Singapore,	" .. .. .	19.58
8. " " " white	" .. .. .	—
9. Penang .. ..	" .. .. .	5.15
10. Singapore ..	" .. .. .	4.48
11. Siam .. ..	" .. .. .	6.72
12. Fine White (as ground for market)	" .. .. .	6.74
13. Finest " "	" .. .. .	5.42
14. Superfine " "	" .. .. .	3.44
15. Long Pepper .. ..	" .. .. .	11.42
16. Another Sample .. ..	" .. .. .	12.96
17. Black Poivrete	" .. .. .	68.80
18. White .. ..	" .. .. .	61.94
19. 10 Black Poivrete 90, No. 3	" .. .. .	16.24
20. 10 White Poivrete 90, No. 9	" .. .. .	8.56

In white peppers the maximum of cellulose seems to be between 6 and 7 per cent, so that more may be learned from these than from the black, where the differences are much greater. I am still, therefore, inclined to look more to the starch, in conjunction with the microscopic examination as an indication of the amount of an organic adulterant.

NOTES ON SENNA.

BY CHARLES HEISCH, F.I.C.

(Read at Meeting, June, 1888.)

HAVING had some samples of powdered senna brought to me by one of my inspectors, I was somewhat puzzled what to do with them. Not only was it possible that other leaves might be powdered with the senna, but that exhausted leaves might be also added. Many varieties of cassia appear to be sometimes found mixed with senna, and so long as you have the leaves you can mostly detect them, but when powdered you lose the characteristic appearances.

The principal adulterants of which I can find any account are *cynanchum argel* and *coriaria myrtifolia*, the latter being a poisonous plant used by dyers and tanners, sometimes called tanners' schumac.

How argel is to be detected in powdered senna, I cannot at present say; I have not yet got a specimen. Fortunately the worst adulterant—the *coriaria myrtifolia* gives when infused precipitates with gelatine, bichloride of mercury, and antim. tart. which senna does not, and also a dark blue with salts of iron. The only bad case of adulteration which I have met with was with buchu leaves. These would have attracted the attention of anyone on the look out, by their different shape and peculiar odour, but in the case referred to did not attract the attention of the purchaser, who made his senna tea, and suffered accordingly. Had the sample been in powder, the mistake would have been



almost unavoidable. As partially exhausted leaves would, of course, give less ash and extract than the senna unexhausted, I made some examinations of undoubtedly pure senna leaves, both Alexandrian and Tinnevely, and though the results show nothing very striking, I think they are of sufficient interest as a small contribution to our knowledge to be worth laying before you.

On examining powdered senna under the microscope, one is struck by the fact that the white translucent hairs from the back of the leaf are quite unchanged by the powdering, so that if one is familiar with the appearance of undoubtedly genuine samples of powdered senna one can get an idea if any other samples contain about the right quantity of hair, which is some guide. I then took the ash in dried samples of the leaves; the amount soluble in water and its alkalinity; the amount sol. in HCl and the insoluble; and, finally, the amount of alcoholic extract calculated on the ash and water-free leaves. The results are contained in the accompanying table.\*

No.	Kind and Source,	Total.	Sol. in Water.	Sol. in HCl.	Insoluble.	Alkalinity as K <sub>2</sub> O	Alcoholic Extract of Ash & Water-free.
1	Tinnevely, Brown and Smart ..	11.48	2.4	8.86	.2	1.16	30
2	Same powdered .. ..	11.22	2.31	8.77	.1	1.14	29.9
3	Tinnevely, Apothecaries' Hall ..	11.34	2.35	8.72	.2	1.16	30.19
4	Same powdered .. ..	11.39	2.67	8.31	.4	1.06	31.78
5	Powdered Alexandrian, Brown and Smart .. ..	11.69	2.35	7.86	1.49	.84	33.3
6	Alexandrian Apothecaries' Hall ..	11.64	2.91	8.36	.37	1.06	29.04
7	Ditto in powder .. ..	11.35	2.66	7.98	.60	2.06	30.13
8	Alexandrian, Allen and Hanbury	12.36	2.96	9.02	.38	1.54	35.5
9	Same powdered .. ..	12.54	3.18	9.12	.24	1.76	35.41
10	Powder from Allen and Hanbury, believed to be mixed ..	13.98	1.22	11.91	.85	1.69	27.75
11	Powder No. 85, from Hampstead	19.01	3.01	12.86	3.14	1.22	29.55
	Ditto No. 88, ditto .. ..	12.89	2.48	9.05	1.36	1.25	30.00
12	Buchu leaves .. ..	6.06	2.73	3.25	0.07	1.47	17.49

It will be observed that the samples obtained from Messrs. Allen and Hanbury contain considerably more ash than the others, and with one exception yield more extract. I have added the results obtained from the two District samples of powder, which in point of extract closely resemble the majority, but one of them differs largely in ash. I have also added the results obtained from buchu leaves, which give about half, both ash and extract.

\* It will be observed that several are done in duplicate, one on the leaf whole and the other on the powdered leaf. I thought it just possible the results might differ, as in the powder the proportion of veins from the leaves might be differently distributed. When bought powdered, the samples mostly contained more ash.

## PEPSIN.

By A. PERCY SMITH, F.I.C., F.C.S., RUGBY.

THE method usually adopted for estimating the peptonising power of pepsina porci consists in dissolving 1 to 2 grains in 8 to 12 ounces of water, to which 40 to 60 minims of hydrochloric acid has been added. 500 to 1000 grains of hard-boiled white of egg, granulated by rubbing through a wire sieve, is immersed in the liquid, and the whole kept at  $98^{\circ}$  to  $130^{\circ}$  F. for four hours, when the undissolved albumen is filtered off through muslin, and, after partial drying, is weighed to ascertain the amount dissolved. The variable numbers above quoted embrace various formulæ recommended by different experimenters.

This method of analysis is excessively crude and untrustworthy. When hard-boiled white of egg is kept in warm water it absorbs a considerable quantity of that menstruum, as much as several units per cent.; consequently, on weighing the residual albumen, you may find that the weight is greater, instead of less than that with which you started, the gain in weight due to absorbed water more than counterbalancing the loss obtaining through solution, as has happened with indifferent samples of pepsin. Then who shall say when, by simple air drying, the albumen has regained its former condition? The enormous quantity of albumen is foreign to the usual habits of the scientific analyst, and involves an enormous waste of time in manipulation.

One trial of this method was enough for me. The first modification I adopted consisted in substituting for the large quantity of granulated albumen a single half of the white of an egg in one piece. I likewise arranged a check experiment in which the pepsin was omitted, other conditions remaining unaltered. At the end of four hours the residual pieces of albumen were placed on blotting-paper to remove superfluous moisture, and weighed. The gain in weight of the albumen in the check experiment, due to absorbed water, was calculated into percentage, and the same deducted from the weights of the other portions which had been subjected to the action of various pepsins. This, although an improvement upon the old method, proved likewise unreliable, because the water absorbed was not equal in each experiment. The albumen which was immersed in acidulated water only quickly dried, superficially, when placed on blotting-paper, whereas that which had been acted on by pepsin was rendered glutinous and incapable of being dried in this manner. In fact one sample weighed considerably more than it did at starting, even after deducting the allowance for water absorbed.

I next tried much smaller pieces of albumen, about 1 c.c., in hope that complete solution might ensue, and a time value be obtained. I soon found, however, that the solubility does not depend upon the mass, but upon the surface exposed.

Finally I discarded altogether the use of fresh white of egg, and had recourse to dry powdered albumen, prepared by drying in a steam oven and levigation in a mortar. With this I succeeded in getting accurate comparisons between the digestive powers of various pepsins. Albumen in this form dissolves with rapidity, owing to its state of fine division. Any remaining undissolved can be filtered off on a counterpoised filter paper, and heated in a water oven until absolutely dry. It is, however, unnecessary to do this when two samples only are compared against each other, nor is it essential to know the actual weight of albumen employed, provided it be the same in each experiment.



This is ensured by placing some on the naked pan of the balance (there is no objection in so doing, as it is a dry gritty powder, and does not adhere to the metal), and counterpoising by a similar addition to the other pan.

Let the albumen fall on the centre of the filtered liquid, avoiding, if possible, contact with the glass of the beaker. It soon sinks, and after the lapse of some time, simple inspection will show which is dissolving with the greater rapidity. Agitation assists solution, therefore take the two beakers, one in each hand, and rotate the contents equally. When one sample has dissolved all the albumen it is manifestly superior to the other which has failed to do so in the given time. If many samples have to be compared it will be necessary to start with known quantities of albumen, and weigh the undissolved residues in the manner above indicated.

An objection may possibly be raised to this modified method, viz., that albumen as suggested is not in the form of a dry powder, and that we ought to copy as nearly as possible the conditions existing in the stomach. To this I would reply that it does not matter in the least, to us, as analysts, what are the conditions which obtain in the stomach; since there is no absolute test for pepsin, we can only compare one sample against another, and that which dissolves the most albumen in the short time is taken to be the best.

Another imperfect method of analysis is that employed in the examination of malt extracts for diastase; in which a certain weight of extract ought to dissolve a certain weight of starch in ten minutes, when if it does so dissolve it, the extract is a good one, and if not it is to be condemned. The more correct way is to ascertain the reducing power of Fehling's solution, before and after digestion with an *excess* of starch, and I intend to say a few words upon this subject on a future occasion, when I have ascertained the maximum amount of diastase existing in the best samples of malt.

#### ON A NEW APPARATUS FOR FRACTIONAL DISTILLATION.

\* BY T. H. NORTON AND A. H. OTTEN.

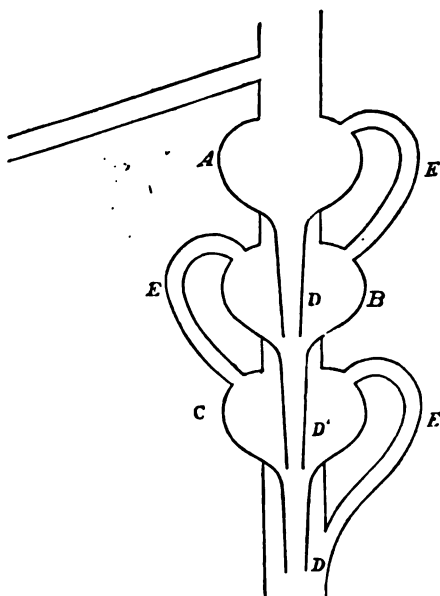
This ingenious apparatus for the technical transformation of ammonium sulphocyanide into calcium sulphocyanide (Wurtz, "Dictionnaire de Chimie, Supplément," p. 597), Dr. J. Scherniac introduced an interesting feature, the so-called *déverseur*, designed primarily to prevent the frothing accompanying the rapid distillation of the ammoniacal liquor. This *déverseur* consisted simply of a cylinder about two-thirds of the size of the retort containing the mixture of lime and solution of ammonium sulphocyanide, and was situated above this retort. The bottom of the cylinder was connected by a pipe with the bottom of the retort, while pipes from the upper portion connected with the top of the retort and with the condensing apparatus. The practical result of this arrangement was that the vapours from the retort entered the top of the cylinder by one opening, and left it by another opening for the condenser, while all liquid carried along mechanically, dropped to the bottom of the cylinder and returned to the retort.

Distillation is carried on so easily and rapidly by means of this device that it seemed a matter of interest to experiment upon the adaptation of this principle of construction to the wants of the laboratory in the form of an apparatus for fractional distillation.

\* American Chemical Journal.

We are indebted to Dr. Tcherniac for a glass model embodying the essential features of the apparatus just described, the details of which will be easily understood from the accompanying cut.

The three bulbs, *A*, *B*, and *C*, are connected with each other by the curved tubes, *E E E*, the lowest connection entering the stem of the apparatus. Small tubes, *D D D*, 2 m.m. in diameter, likewise project from the bottom of each bulb. Their capillarity is increased by passing a platinum wire through the entire series. When the device is inserted into a distilling flask, it will be seen at once that the current of vapour takes a zig-zag course, passing through the tubes, *E E E*, and across the top of each bulb, while all condensed liquid returns directly by the route of the tubes *D D D* into the distilling flask. A fractional distillation performed by means of this apparatus would hence be



conducted on principles directly contrary to those which underly the construction of our ordinary technical and laboratory stills, in which the central object is to bring the ascending current of vapour into as intimate contact as possible with the downward flow of liquid resulting from the condensation of less volatile constituents.

In order to test the availability of the new design we brought it into direct comparison with two well known types of fractionating apparatus, viz., those of Hempel and of Linnemann, both based on the ordinary principle of a thorough washing of the vapour current by the products of partial condensation.

The Linnemann apparatus used consisted of a tube, 1 c.m. in diameter, with three bulbs blown along a space of 16 c.m., and contained three cups of platinum gauze. The Hempel apparatus consisted of a similar tube, the cups being replaced by a layer of glass beads, 7 c.m. deep.

The new device contained three bulbs blown on a tube 1 c.m. in diameter, along



pace of 12 c.m., as described above. Comparative fractional distillations were conducted with several mixtures of known quantities, every precaution being taken to order the conditions identical in the different series. In all cases 100 c.c. of liquid were used, and the fractions were collected at intervals of 3° in graduated cylinders. After each addition, during the fractionation, of a new quantity of liquid, fractions were collected during the two preceding temperature intervals.

The quantities given are in cubic centimetres. The first column, A, gives the results obtained with the Linnemann apparatus; the second column, B, those with the Hempel apparatus, and the last column, C, those with the new apparatus.

## FIRST SERIES.

Mixture of 50 c.c. of acetone (96 per cent.), boiling point 56°-57°, and 50 c.c. of ethyl alcohol, boiling point 66·6°.

<i>First Fractionation.</i>				<i>Second Fractionation.</i>				<i>Third Fractionation.</i>			
	A.	B.	C.		A.	B.	C.		A.	B.	C.
59°	31	40	33	56-59°	46·5	53	47·5	56-59°	51	58·5	50·5
62	46	38	39	59-62	28	24·5	23·5	59-62	19·5	18·5	18
65	14·5	12·5	16·5	62-65	8	9·5	11	62-65	6·5	8	7·5
67	8	8·5	11	65-67	14·5	11·5	16·5	65-67	18	13	20
	<u>99·5</u>	<u>99</u>	<u>99·5</u>		<u>97</u>	<u>98·5</u>	<u>98·5</u>		<u>95</u>	<u>98</u>	<u>96</u>
<i>Fourth Fractionation.</i>				<i>Fifth Fractionation.</i>							
	A.	B.	C.		A.	B.	C.				
56-59°	57	61·5	55	56-59°	58	63	55·5				
59-62	11·5	13	13·5	59-62	9·5	12·5	12				
62-65	6·5	7·5	6	62-65	5	5·5	4·5				
65-67	18	14·5	21	65-67	20	14·5	24·5				
	<u>93</u>	<u>96·5</u>	<u>95·5</u>		<u>92·5</u>	<u>95·5</u>	<u>96·5</u>				

## SECOND SERIES.

100 c.c. crude wood alcohol.

<i>First Fractionation.</i>				<i>Second Fractionation.</i>				<i>Third Fractionation.</i>			
	A.	B.	C.		A.	B.	C.		A.	B.	C.
66°	48	45·5	46·5	63-66°	57	50·5	52·5	63-66°	60	56·5	61
69	30·5	33	31·5	66-69	26	30	27	66-69	24·5	30	21·5
72	12·5	16·5	13	69-72	6	12·5	11·5	69-72	6·5	6	8
75	4	1·5	6·5	72-75	3·5	1·5	4	72-75	2	1	2
+	2·5	1	2	75+	3·5	0·5	3·5	75+	3	0·5	4·5
	<u>97·5</u>	<u>97·5</u>	<u>99·5</u>		<u>96</u>	<u>95</u>	<u>98·5</u>		<u>96</u>	<u>94</u>	<u>97</u>
<i>Fourth Fractionation.</i>				<i>Fifth Fractionation.</i>							
	A.	B.	C.		A.	B.	C.				
63-66°	70·5	64·5	71	63-66°	74·5	65·5	75·5				
66-69	16	21·5	14	66-69	13	20·5	11				
69-72	3·5	6·5	5·5	69-72	3·5	6	5				
72-75	2	1	2	72-75	1	1	1				
75+	3	0·5	3·5	75+	3	0·5	3·5				
	<u>95</u>	<u>94</u>	<u>96</u>		<u>95</u>	<u>93·5</u>	<u>96</u>				

## THIRD SERIES.

Mixture of 50 c.c. of ethyl alcohol, boiling point 78.3°, and 50 c.c. of water, boiling point 100°.

<i>First Fractionation.</i>				<i>Second Fractionation.</i>				<i>Third Fractionation.</i>			
	A.	B.	C.		A.	B.	C.		A.	B.	C.
79-82°	27	29.5	27	79-82°	33	42.5	39	79-82°	43.5	46	43
82-85	16.5	16.5	18	82-85	13.3	7	8.5	82-85	4.5	4.5	6
85-88	8	8.5	6.5	85-88	4.5	4	5	85-88	4	3	3.4
88-91	5	5	5	88-91	4.5	2.5	3.5	88-91	0.5	1	2
91-94	6	3.5	4	91-94	1	1.5	2.5	91-94	0.5	0	0.4
94-97	3.5	3.5	4.5	94-97	1.5	1	1.5	94-97	0.5	1	0.4
97-100	32	31.5	32	97-100	39.5	38	38	97-100	41.5	41	41
	98	98	97		97.3	96.5	98		95	96.5	96.4

<i>Fourth Fractionation.</i>				<i>Fifth Fractionation.</i>			
	A.	B.	C.		A.	B.	C.
79-82°	46.5	49	47	79-82°	50	51	49
82-85	4	3	4	82-85	1.5	2	3.5
85-88	2	2	2.5	85-88	0	0.5	0
88-91	0	0	0.5	88-91	0	0	0
91-94	0	0	0	91-94	0	0	0
94-97	0.5	1	0.5	94-97	2	2	1.5
97-100	42.5	41	42	97-100	43	40.5	42.5
	95.5	96	96.5		96.5	96	96.5

## FOURTH SERIES.

Mixture of 50 c.c. of toluene, boiling point 110°, and 50 c.c. of amyl alcohol, boiling point 131°.

<i>First Fractionation.</i>				<i>Second Fractionation.</i>				<i>Third Fractionation.</i>			
	A.	B.	C.		A.	B.	C.		A.	B.	C.
108-111°	40	42.5	43	108-111°	49	42.5	47	108-111°	50	44	50
111-114	20.5	19.5	15.5	111-114	8.5	11.5	10	111-114	6	9.5	7
114-117	6	9	9.5	114-117	4	7	6.5	114-117	3.5	5.5	4
117-120	2	4	5.5	117-120	3.5	5	4	117-120	2.5	4	2
120-123	2	5	4.5	120-123	3	4	3.5	120-123	2.5	4	2
123-126	5	4	4.5	123-126	3.5	4	3	123-126	1.5	3.5	4
126-129	22.5	14	16	126-129	26	23	23	126-129	31	27	26
	98	98	98.5		97.5	97	97		97	97.5	96

<i>Fourth Fractionation.</i>				<i>Fifth Fractionation.</i>			
	A.	B.	C.		A.	B.	C.
108-111°	52	48.5	53	108-111°	53	47.5	55
111-114	4	5.5	3.5	111-114	3.5	7.5	3
114-117	2.5	7	3	114-117	3	3	2.5
117-120	2	1.5	3	117-120	0.5	4	1.5
120-123	1.5	3	3.5	120-123	0.5	1	1
123-126	1.5	3	1.5	123-126	2.5	2.5	1.5
126-129	33	29	2.75	126-129	34	31	31
	96.5	97.5	95		97	96.5	95.5



## FIFTH SERIES.

Mixture of 50 c.c. of benzene, boiling point  $80.5^{\circ}$ , and 50 c.c. of toluene, boiling point  $110^{\circ}$ . (In this series the Linnemann tube (A) was provided with six platinum gauze cups, instead of three, as in the preceding series.)

<i>First Fractionation.</i>				<i>Second Fractionation.</i>				<i>Third Fractionation.</i>			
	A.	B.	C.		A.	B.	C.		A.	B.	C.
81-84°	30	25	20	81-84°	32	37	28	81-84°	38	39	32
84-87	8	10.5	13	84-87	10.5	7	6.5	84-87	3.5	6	5
87-90	11	8	7	87-90	5	6.5	6.5	87-90	5	4	6
90-93	5	10.5	10	90-93	5	4	6.5	90-93	4	3.5	5.5
93-96	4	4	6.5	93-96	3	1	6	93-96	3	1.5	4.5
96-99	3	5	8.5	96-99	2.5	3.5	5	96-99	2	2	2
99-102	3	4	5	99-102	2	4	4.5	99-102	1	3	3.5
102-105	7.5	5.5	5	102-105	4.5	5.5	6.5	102-105	2	4.5	4.5
105-108	10	9	7	105-108	6	6.5	7	105-108	4.5	5.5	6.5
108-111	13	14.5	14	108-111	21	20	18	108-111	27	23	24
	94.5	96	96		91.5	35	94.5		90	92	93.5
<i>Fourth Fractionation.</i>				<i>Fifth Fractionation.</i>							
	A.	B.	C.		A.	B.	C.				
81-84°	41	41.5	34.5	81-84°	43	44.5	37				
84-87	4	4	5	84-87	3	3.5	3.5				
87-90	4	3	4.5	87-90	2	2.5	3.5				
90-93	3	4	4.5	90-93	1	1	2.5				
93-96	2.5	2	2.5	93-96	0	1	2.5				
96-99	0.5	2	3.5	96-99	2.5	1	2				
99-102	1	1	2.5	99-102	0	1.5	1.5				
102-105	2.5	2.5	3.5	102-105	2.5	2.5	2.5				
105-108	3.5	4	4	105-108	5.5	3.5	5				
108-111	29	26	27	108-111	28	28.5	28.5				
	91	90	91.5		87.5	89.5	88.5				

These experimental data are of interest as showing that no one form of apparatus is superior to other forms for all cases. Thus in the series of distillations of mixtures of acetone and methyl alcohol, larger fractions of the latter constituent are separated by the use of the new apparatus than by the use of the other two forms. Hempel's apparatus yields a larger fraction at the boiling point of acetone in the above case than either of the other forms, while, on the contrary, in the case of wood spirit, its initial and terminal fractions are both inferior in amount to those of the other forms. Again, in the distillation of a mixture of alcohol and water there appears to be but slight difference in the effects obtained by the three forms. In the case of a mixture of benzene and toluene, the two older structures permit the separation of decidedly larger benzene fractions, while the toluene fractions are about the same in all cases. When the toluene is mixed with amyl alcohol, the new apparatus gives better results with the lower fraction than the other two forms, while it is behind them in the separation of the alcohol.

As far as the power of rapid fractionation is concerned, the new form of apparatus, while giving results quite as satisfactory as those yielded by the two types hitherto regarded as the most efficient, certainly does not exceed them in the average thoroughness of separation.

With regard to the simplicity of construction Hempel's apparatus still remains unsurpassed, while the lateral tubes of the new form render this type certainly more fragile than the original Linnemann apparatus, and place it rather in the category of such a device as that of Le Bel and Henninger.

Although the new type cannot claim advantages which should lead to its superseding the present forms of apparatus for fractional distillation, the experiments made with it show conclusively that the principle involved in its construction, of the rapid separation of vapour and condensed liquid, yields at least as satisfactory results in practice as the totally opposite principle of a continuous washing of vapour in condensed liquid.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

PHYSIOLOGICAL EFFECT OF SAFFRON SUBSTITUTE OR POTASSIC DINITRO-CRESYLATE. VAL. GERLACH.—*Zeitschr. f. angew. Chemie.* No. 10.—This body,\* now so largely used for colouring mustard and farinaceous foods, is of a decidedly poisonous nature. The author experimented on rabbits, cats, and dogs. Of these experiments a few will no doubt be interesting to analysts. Experiment No. 4.—Rabbit weighing 1,750 grammes. .25 of the compound is administered. After 15 minutes, difficulty in breathing; after 25 minutes, passes intensely yellow-coloured urine, and motions become very uncertain; after 30 minutes, its head hangs down, and the extremities get stiff; after 33 minutes, 60 difficult respirations per minute; after one hour, death. *Post-mortem*—stomach very yellow, its mucous membrane peels off, brain hyperæmic. Experiment No. 5.—Dog weighing 11,200 grammes: 2.8 grammes of the compound are administered in a piece of meat. After a few minutes vomiting sets in, but the animal again swallows it. Otherwise it is all right and plays about, 23 respirations per minute; after 40 minutes, the animal rejects all its food, is very restless, and has many liquid stools; 132 respirations per minute. Violent cramps all through the body, and it seems to suffer constant fright; after 50 minutes, strong attack of cramp in the extremities; it throws itself on its back, and bends its head. The tongue has lost its yellow colour, attack passes off after 10 minutes, and it lies still, but after another 7 minutes another attack comes on, and the tongue protrudes; still conscious, and attack again passes off in 5 minutes; after 1 hour and 17 minutes, the dog suddenly jumps up and stares at a spot on the wall as if it had a vision. It now falls again suddenly on its back with legs doubled up; after 1 hour and 39 minutes, the animal jumps up in a great fright, but 7 minutes later it is on the road to recovery. Experiment No. 6.—A kitten eats some of the intestines of the poisoned rabbit and suffers from similar symptoms as the dog. Death occurred after 10 hours. *Post-mortem*—stomach and intestines yellow, vessels strongly injected, brain hyperæmic. Experiment No. 11.—Action of small continued doses. A dog weighing 7,120 grammes is given .1 gramme of the compound dissolved in water but mixed with its ordinary food. Notwithstanding it had been without food for 24 hours it refused to touch the mixture even after  $3 \times 24$  hours. A second dog proving equally obstinate, the first dog, now weighing only 7,100 grammes, receives daily .1 gramme of the compound wrapped up in a piece of meat, which it swallows, and then

\* For distinguishing between this body and Welter's bitter, see ANALYST, January, 1897.



the usual amount of ordinary food is given. After 29 days, when it has become much thinner, the animal is suddenly seized with spasms, and similar symptoms as already described are noticed, and after 48 hours it dies. *Post-mortem*—weight of body, 5,950 grammes. Fat nearly disappeared, muscles still fairly well developed. The stomach contains besides undigested matter, a thickish blackish-brown mass chiefly consisting of blood, no doubt caused through an ulcerated state of the mucous membrane. Kidneys normal, brain hyperæmic.

Surely the use of this colouring matter should not be allowed.

L. DE K.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

DETECTION OF SEED OILS IN OLIVE OIL. M. BRULLE. *Comptes Rendus*, April, 1888.

—Place into a test tube 10 c.c. of the oil to be examined, and add .1 gram. of dried and powdered albumen, and 2 c.c. of nitric acid. Heat until the acid boils, and then incline the tube so that the ebullition shall cause the oil and the albumen to mix together. If the olive oil is pure it will become faint greenish-yellow, but if it contain a notable proportion of seed oil the colour will be deep orange-yellow. Even five per cent. of such oil will cause a distinct amber-yellow tint. This test is applicable to the admixture of cotton, ground nut, sésame, colza, and in fact all oils derived from seeds, excepting only an oil known in French commerce as *aveline exotique*, which latter gives a rose tint with whitish spots. The test works with nitric acid only if the adulterating oil be coloured, but with colourless oils like ground nut, etc., the addition of the albumen is necessary, and it is in this that the novelty of the process consists. The limit of delicacy is 5 per cent. of adulteration.

W. H. D.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

DETERMINATION OF MORPHINE IN OPIUM. A. KREMEL. *Chemiker Zeitung*.—Five grammes of the opium powder are macerated in a small flask, with 75 c.c. of lime water, for 12 hours, with frequent shaking; this is then filtered through a plaited filter. (The ratio 1 part of opium to 15 parts officinal lime water has been arrived at by practice, and in this way there is obtained a nearly neutral or slightly acid filtrate. Should the liquid after the maceration react alkaline, then less lime water should be used.) To 50 c.c. of the filtrate, corresponding to 4 grammes of opium, which is brought into a weighed flask of such a size as to be nearly filled by the ether and ammonia, there is added 15 c.c. of ether and 4 c.c. of liquor ammonia. The flask is then well corked and the contents mixed. The flask is then set aside for 6 to 8 hours, the temperature being kept at 10°–15°. At the end of that time the ethereal layer is removed, 5 c.c. of fresh ether added, and the flask gently shaken; the ether is again removed, and finally the crystals of morphine, which have separated out, are collected on a small plaited filter. The crystals which remain in the flask are washed with 5 c.c. of distilled water; this wash-water is brought on the filter, and finally the flask, the filter, and its contents are dried at 100°. The crystals on the filter are transferred to the flask, and this is then dried until a constant weight is obtained. The morphine thus obtained is perfectly pure, and dissolves *completely*, though slowly, in 100 parts of lime water. The mode of procedure is the same for opium extract, 1 part of extract is treated with 20 parts of lime water. Opium tincture is first mixed with powdered glass which has been washed with hydrochloric acid, and the mixture evaporated to dryness on the water-bath; the residue is then treated with a quantity of lime water equal to the quantity of tincture used in the experiment.

W. H. D.

## LAW NOTES. TO OUR READERS.

*Some years ago it was decided to discontinue the reporting of ordinary police-court proceedings, and only to give such cases as authoritatively established some point in connection with the working of the Acts in which public analysts are interested. After a fair trial of this system, a majority of the members of our Society have expressed a wish that our old practice should be, to some extent, returned to, and, in deference to such request, we have decided to resume the reporting of police proceedings. The line will be drawn at accounts of ordinary cases, and reports will only be inserted when any novel, legal, or chemical point arises, or where the certificate of the analyst is in any way attacked. Any member or subscriber connected with such a case is, therefore, invited to furnish us with a report of the proceedings, signed with his name, not for publication, but as a guarantee of exactitude.*

**THE LABELLING OF MARGARINE.**—In the Glasgow Sheriff Court yesterday, Sheriff Balfour gave judgment in a prosecution in which Mrs. Ann Martin, or Dowall, provision merchant, 93, Centre Street, Tradeston, was charged with having, on 23rd ult., exposed margarine for sale by retail without having attached to each parcel a label marked "Margarine" in printed capital letters not less than 1½ inches square. On 12th inst., when the case was first brought before the Court, Mr. Angus Campbell, writer, tendered a plea of not guilty on behalf of the respondent. After hearing evidence his lordship continued the case until yesterday, when he gave judgment. The Sheriff said that the Sanitary Officer went into the shop and found on a back shelf behind the counter a kit of margarine, which the authorities maintained was exposed for sale by retail, and ought to have had attached to it a label marked "Margarine" in printed capital letters not less than 1½ inches square. The foundation of the prosecution was that the package or kit was virtually a parcel exposed for sale by retail. It was contrary to the meaning of the section to hold that the kit was a parcel, as he considered that the authorities, in framing that section, meant to deal with the kit as a package, and with portions of its contents as parcels. The sanitary officer did not see any margarine delivered out of the package, and he himself did not purchase any margarine out of it, but he was told by some person in the shop that they were selling from it. But, the Sheriff said, he could not hold that because a kit was on a back shelf, properly branded in terms of the Act, and by reason of a sanitary officer going into the shop and ascertaining in this rough and round way that they were selling from it, without giving any details of the sales, that therefore the person was contravening the Act of Parliament, and that she ought to have had the package labelled as a parcel. His lordship dismissed the case.

The Sheriff afterwards gave judgment in the case of John Masterton, provision merchant, 24, Barnack Street, who was charged with a similar offence. A piece of cloth was found partly concealing the brand, but the Sheriff said he could not hold that defendant had purposely done this, and therefore dismissed the case.

John Hill, provision merchant, 51, Prince's Street (City), was fined 30s. for exposing two moulds and a box filled with lumps of margarine, which were unlabelled.

**SALICYLIC ACID IN BEER.** MONTHLY REPORT OF THE FOOD ANALYSTS IN AMSTERDAM. —"Of beers thirty-four different kinds were tested for salicylic acid. On applying the extraction process of Röse, which enables us to detect as little as .0001 gramme of salicylic acid in one litre, we found every sample to be perfectly free from this substance. If, however, the results of the distillation process are to be taken as correct, every one of the samples would have been adulterated. We are of opinion that beer contains a volatile substance, or at least, that on distilling with sulphuric acid a substance giving salicylic acid reaction is produced."

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

To the Editor of the ANALYST.

SIR,—In answer to Professor Asboth's paper on "The Estimation of Starch," which appeared in this month's ANALYST, in which he says he does not agree with me in saying that the composition of the compound formed by baryta on starch varies, I think if Professor Asboth follows directly his own instruction of simply standardising his baryta, and not making sure that it is in excess, he will find that it varies considerably.

In my paper I recommended the baryta to be used of the strength capable of saturating 90 cc. N/10 HCl, which strength I found was in excess, and therefore always gave the required compound.—

am, yours truly,

Edinburgh, July 26th, 1888

J. NAPIER SPENCE.



# THE ANALYST.

SEPTEMBER, 1888.

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### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual country meeting was held on Friday, July 27th, at Sheffield. During the day, the members and their friends visited, under the guidance of the President, the German Silver, Britannia metal, and electroplating works of Messrs. Walker and Hall, the cutlery works of Messrs. Joseph Rodgers and Sons, the steel works of H. Bessemer and Co., and also the Atlas Works of Sir John Brown and Co., Limited, where they witnessed various interesting metallurgical operations, including the manufacture of steel by the Siemens-Martin process, the rolling of tyres for railway wheels, etc.

In the evening the party dined at the Royal Victoria Station Hotel, after which the general meeting was held, and the following papers were read and discussed:

"On the use of the term 'Normal' in Volumetric Analysis." By the President.

"On the Examination and method of stating the Results of the Analysis of Boiler-Waters." By the President.

"Note on the Examination of Lard." By Thos. S. Gladding (New York).

"Water Reports—A Dilemma." By M. A. Adams, F.R.C.S.

Mrs. Allen afterwards held an At Home, which was attended by all the members of the party.

On the following morning the party drove, via Fox House and Froggatt Edge, through Baslow to Chatsworth House, the seat of the Duke of Devonshire, after viewing which they proceeded to the Chatsworth Hotel, Edensor, where luncheon was provided, and thence to Haddon Hall, and on to Bakewell, and so by train to Buxton.

Unfortunately the weather interfered with the excursions which had been arranged for in the Peak District, but on the whole the gathering was a very enjoyable one.

### ON THE DETECTION OF COTTON-SEED OIL IN LARD.

BY ALFRED H. ALLEN.

(Read at Meeting, June, 1888.)

I HAVE recently had occasion to look into the question of the adulteration of lard with cotton-seed oil, and for the purpose of comparison have analysed certain samples, known

and suspected to be adulterated, together with certain specimens of which the genuine nature was beyond suspicion.

The following table shows some of the results obtained. The figures given by the lard marked "Omentum" were yielded by a specimen rendered in my laboratory :—

	Omen- tum Lard.	Leaf Lard.	English Lard.	American Lard containing Cotton-seed Oil.	Mixture of unknown nature.	Suspected sample.
ORIGINAL FAT.	Melting point; °C .....	39.0	40.0	39.0	37.5	40.0
	Solidifying point; °C .....	26.5	32.0	27.0	27.5	30.5
		rising to 27.5				
	Plummet gravity at 99°C .....	.8602	.8620	.8608	.8648	.8637
FATTY ACIDS.	Iodine absorption, per cent. ....	55.4	60.5	62.0	82.5	68.8
						63.8
	Melting point; °C .....	39.0	39.5		39.5	
	Solidifying point; °C .....	38.7	38.5		37.5	
		rising to 39.0	rising to 38.8		rising to 38.5	
	Plummet gravity at 99°C .....	.8372	.8385		.8450	.8385
	Mean combining weight .....	274.5			276.8	
	Iodine absorption, per cent. ....	58.3	65.3		70.4	64.8
	Oleic acid, etc., per cent. ....	58.4			57.8	
	" " Iodine absorption .....	87.4			(94.6)	
	Milliau's nitrate of silver test .....	white	white	grey	Marked black- ening.	Sensible dark- ening.

The following figures are the recorded results of experience with tallow, lard, and cotton-seed oil :—

	Tallow.	Lard.	Cotton-seed Oil.
ORIGINAL FAT.	Melting point; °C .....	—	28—45
	Solidifying point; °C .....	33—48	fluid
	Specific gravity at 99°C .....	.862	.860—861
	Iodine absorption, per cent. ....	40	59—62
FATTY ACIDS.	Melting point; °C .....	45	35—36
	Solidifying point; °C .....	43	30
	Specific gravity at 99°C .....	—	846.7
	Iodine absorption, per cent. ....	41.3	64.2

In addition to these Müller has recorded the following iodine-absorptions :—Beef-suet, 38.4; lard, 55.0; cotton-seed oil, 107.9.



Mr. J. Carter Bell informs me that he recently found 51·5 as the iodine-absorption of a sample of leaf-lard which he prepared himself. The same sample yielded 51·2 in the hands of Mr. R. Williams, while Mr. E. W. T. Jones found 46·57.

I have this day received from Mr. Chas. M. Blades a sample of cotton-seed stearin, which is now a commercial product. I have attempted in vain to obtain this article for years past, in consequence of the result recorded by Dr. Muter (*ANALYST*, vii., 93), who described the adulteration of lard with this product. The *ordinary* cotton-seed stearin of commerce is really the stearic acid from cotton-seed oil, and quite a different article from the sample of neutral fat now before you. The figures given in the following table, in the column headed "Cotton-seed Fat," are those yielded by the sample in question :—

	A. Cotton-seed Fat.	B. Cotton-seed Oil.	C. Cotton-oil Acids from B.
Plummet gravity at 99°C.....	·8684	·8725	·8476
Melting point.....	40°C	—	35°C
Solidifying point .....	31 rising to 32·5°	—	32°
Iodine absorption .....	89·8	108—110	115·8
Saponification equivalent .....	—	285—294	289*
Acidity (= Oleic Acid).....	·34	trace	97·6

The results I have obtained substantially confirm those of Mr. Jones, Mr. Rowland Williams, and other previous observers, and appear to show that lard differs materially in its iodine-absorption from beef-stearin on the one side and cotton-seed oil on the other. There is also a marked difference in the specific gravity of lard and cotton-seed oil, and this difference is also noticeable in the fatty acids. On the other hand, lard and beef-fat are substantially of the same density. This difference is very important, as it would enable one to distinguish a mixture of beef-stearin and cotton-seed oil, having an iodine-absorption of about 60, from genuine lard. Thus, while the proportion of the adulterant in a mixture composed of lard and cotton-seed oil *only* can be ascertained with considerable accuracy by determining the iodine-absorption, the estimation will be below the truth if beef-stearin be present. On the other hand, the presence of beef-stearin does not interfere with the deduction to be drawn from the increased specific gravity of the melted sample. Hence this method, though not affording more than approxi-

\* Mr. Rowland Williams, in his recent paper on the characters of certain fatty acids (*ANALYST*, xlii., 89), quotes "other observers as recording a range of 275 to 337 for the mean combining weight of the fatty acids from cotton-seed oil." On reference, I found, to my surprise, that the anomalous figure 337·2 was one recorded by myself. I feel sure this figure is erroneous, though I am unable, at this distance of time, to ascertain the cause with certainty; but the specimen said to have that combining weight was of considerable age when analysed.

mate results, is calculated to do very good service in conjunction with the iodine-absorption.

As a qualitative test, I have found Milliau's modification of Becchi's nitrate of silver reaction to be very valuable. It is quite possible that it may be used for obtaining rough quantitative results, but I have not had sufficient experience of it in this direction to express a positive opinion. Milliau's test certainly seems to be a great improvement on that originally proposed by Becchi, in which rape-oil and amylic alcohol were used with no obvious purpose.

My experiments did not indicate that either the melting point or solidifying point of the original sample, or of the fatty acids therefrom, is capable of affording any useful information.

The iodine-absorption of a sample of lard-oil was found by Mr. Rowland Williams to be 73 (private communication). A small quantity of lard-oil which I obtained by pressure from lard of known purity showed an iodine-absorption of 74. On the other hand, a sample of lard-oil several years old, believed to be pure, gave only 41, and Müller has recorded an iodine figure of 47.2.

It will be observed that I have determined the iodine-absorptions of the fatty acids of some of the samples, in addition to making the determinations on the original fats. This plan has been recommended by Rose, and Mr. R. Williams has recently recorded some experiments made in the same way. There is one important practical advantage in operating on the fatty acids, and that is that in consequence of their solubility in alcohol, the use of chloroform can be entirely dispensed with. Chloroform is liable to have more or less reducing action on Hübl's iodine solution, and hence a quantity equal to that used in the test must be employed in the blank experiment for ascertaining the strength of the iodine solution. If, as sometimes happens, more chloroform has to be added during the analysis to maintain proper solution, an uncertainty is introduced, unless another blank experiment be made with a larger quantity of chloroform. All this is avoided if the fatty acids be employed instead of the original fat, and the chloroform wholly omitted. The fatty acids are best prepared by saponifying the fat with rectified spirit (not methylated), and the saturated aqueous solution of caustic soda, recommended by Wollny for use in Reichert's butter process.\* A perfectly colourless product is thus obtained.

I may take this opportunity of cautioning employers of Hübl's iodine process, of the importance of adhering strictly to the prescribed process, unless the modification adopted be fully described and its effects allowed for. Already there are indications of deviation in detail in manipulation which are liable to cause much trouble and confusion at a moment when analysts are trying to ascertain within what range the absorptions of lard, cotton-seed oil, and other fats are liable to vary.

I have made some experiments with a view of isolating from adulterated lard the fatty acids forming lead salts soluble in ether. The high iodine-absorption of cotton-seed oil being presumably due to the presence of the glyceride of linoleic or similar acid, it would seem to follow that the iodine figure should be higher after the separation of

\* I have been using Wollny's modified butter method extensively, and with the most satisfactory results. The improvement in sharpness and certainty amply compensates for the little extra time and trouble involved.



the acids of the stearic series. My experiments in this direction have not been very satisfactory, and the tedious nature of the process is such as to discourage one from following up the matter, unless absolutely necessary. I think that the experience we now have shows that cotton-seed oil can be detected with certainty in lard, even when present in very moderate quantity, if it be the sole adulterant. The determination can, at present, only be affected with tolerable accuracy in the absence of beef-stearin. The detection of cotton-seed oil is quite easy, in any case, but the determination becomes more uncertain if beef fat be present. From the combined results of the determinations of iodine-absorption and specific gravity it would be possible to form an idea of the proportion of beef-stearin present in a mixture of that substance with lard and cotton-seed oil.

Of course it is necessary to multiply results and to avoid the error of laying down, at this stage, too rigid limits of variation; but the tools we now possess, if used skilfully, appear to be amply sufficient to solve the problem presented to us.

I have to thank Mr. W. Chattaway and Mr. W. H. Barracrough for their zealous co-operation and assistance.

#### ON MIXED LARD AND THE DETECTION OF COTTON-SEED OIL.

BY OTTO HEHNER.

(*Read at the Meeting, June, 1888.*)

A BILL has lately been laid before the United States House of Representatives to Regulate the Manufacture and Sale of Counterfeit or Compounded Lard.

From the memorial to the Senate and House of Representatives in support of the Bill I take the following interesting details:—

"The annual production of lard in the United States is now estimated at 600,000,000 pounds, of which more than 35 per cent. is an adulteration. Lard is one of the great staple products of the country."

"The advocates of the proposed legislation do not object to the use of cotton-seed oil as food, either alone or in combination with other substance; they make no war upon that article. They do not object to the manufacture and sale of any new and healthful article of food; but they do object to the sale of compounds as *lard* and the concealment of the fact that they are *not lard*."

They define lard as "the fat of swine after being melted and separated from the flesh," "not any particular part of the fat, but whatever is the fat of swine thus treated is lard; that is the generic name."

In 1886-7, 17 million hogs went to market, the lard from which alone amounted to more than 527 million pounds, representing a value of 40 million dollars. More than 325 million of pounds of it were exported to foreign markets, bringing into the United States nearly or quite 30 million dollars."

"Forty years ago *refined* lard came largely into use. It was made by pressing out of pure lard a portion of the oil, the lard stearine being left behind. This was added to unpressed lard for the purpose of stiffening it, the mixture being called *refined lard*, consisting entirely of the fat of swine. It commanded for 40 years a higher price

than ordinary lard, the difference being on the average one half cent in favour of the refined article."

"During the last seven or eight years compounds have been made consisting of lard, cotton-seed oil, and beef stearine; or lard, water, cotton-seed oil, and beef stearine; or cotton-seed oil, beef stearine, and no lard, or only a very faint trace of lard. These compounds were sold as 'refined lard,' 'pure refined lard,' 'choice refined family lard,' and other similar names."

Professor H. W. Wiley, in his evidence, states that "he analysed 12 samples of Fairbank's manufacture, all of which contained cotton-seed oil, eight being branded 'Prime refined family lard,' one 'Choice refined family lard,' one 'Pure refined family lard,' one 'Prime refined family lard,' and one only 'Compound lard.' Of 12 samples of Armour and Co.'s manufacture ten contained cotton-seed oil, two being pure lard."

"Messrs. Armour and Co. state that they use from 59 to 75 per cent. of lard in their mixtures, Fairbank from 50 to 75 per cent. The former firm use 75,000 barrels of cotton-seed oil, and make 60 million pounds refined lard. 75,000 barrels weigh 28,125,000 pounds; hence the average proportion would be 46 per cent. of cotton-seed oil in the mixture. But as from 10 to 15 per cent. of stearine are necessary to stiffen the mixture, the proportion of lard would be only about 42 per cent."

Abundant evidence was collected in proof of the above statements, but I would take exception to the statement that lard is the fat of swine generally. Of late years certainly the whole of the fat has been used in the manufacture of lard, but originally only the belly fat was employed. This is the true source of lard.

The test relied upon by Professor Wiley for the detection of cotton-seed oil is the nitrate of silver test, presumably that described by E. Bechi. This has, since its publication in 1884, been variously modified. Bechi dissolves 1 grain of silver nitrate in 200 c.c. of 98 per cent. alcohol, and adds 40 c.c. ether. He also makes a solution of 15 per cent. of rape oil in alcohol. 10 c.c. of the oil to be examined are mixed in a flask with 1 c.c. of the silver solution, and 12 c.c. of the alcoholic rape oil solution; the whole is well shaken and heated for a quarter to half an hour upon the water bath. In presence of cotton oil the mixture assumes a dark colouration. The Italian Government at once appointed a commission to investigate the method. The report is to the effect that the method is perfectly trustworthy, provided the reagent contains a small quantity of pure nitric acid, and the oils to be tested have previously been filtered. The reagents recommended are composed as follows:—

1. Silver nitrate, 1 grm.  
Alcohol, 200 grms.  
Ether 40 "  
Nitric acid, .1 grm.
2. Amylic alcohol, 100 grms.  
Colza oil 15 "

Fifteen per cent. of cotton oil can readily be detected in olive oil, but if less than 10 per cent. are present the results are doubtful. If too little free acid be present, reduction may take place even in absence of cotton oil; if too much, no reduction takes place even with cotton oil.



Milliau (*ANALYST*, xiii., p. 95), instead of testing the oils direct, separates the fatty acids, takes about 5 c.c. and heats with 2 c.c. of a 30 per cent. aqueous solution of nitrate of silver. In this manner he claims to have easily detected a one per cent. adulteration.

I have made many experiments with pure and mixed lards, and can fully corroborate the Italian Commission as to the importance of the presence of a small quantity of free nitric acid, but Bechi's test can, without impairment of its delicacy, be simplified by the omission of the amylic solution of colza oil. I make a solution of nitrate of silver in alcohol and ether, *very* slightly acidified, and add to the oil to be examined about half of its bulk of the silver solution, then heat on the water bath for a quarter of an hour, no longer. Pure lard always remains perfectly unchanged, cotton oil mixtures blacken more or less quickly. It is quite possible to arrive at approximately quantitative results, by comparing the oil mixtures of known composition. I consider the test an invaluable one. It is also quite applicable to butter and margarine. Pure butter does not reduce the silver solution; margarine, containing as it does almost invariably some cotton oil, reduces strongly. I can see no advantage in Milliau's modification, especially as it renders the test a much more complicated one.

As to the quantitative estimation of cotton oil in lard, the Mauméné reaction, if properly applied, answers all requirements. It is essential that the sample to be tested be free from water. If the lard does not melt quite clear, it must be heated over a very small naked flame, until all water is driven off.

Fifty grammes of pure lard with 10 c.c. of strong sulphuric acid show a rise of from 24 to 27.5 degrees Centigrade, cotton oil of about 70 degs. Mixtures made by myself yielded increase of temperature as nearly as possible corresponding to the theory. In every case a lard which reduces silver shows a rise higher than 27.5 degs. Thus I have examined samples which produce a rise of 29, 35, 36, 38.5, 40, and 41.5 degs., the first sample reducing silver slightly but unmistakably.

If in any doubtful case further corroboration is required, the method lately described by E. Salkowski (*Zechl. Anal. Chem.* xxvi., 557) is extremely useful. Salkowski has shown that, whilst animal fats contain invariably a small quantity of cholesterin, vegetable fats contain an allied substance, phytosterin. The former crystallises from alcohol in flat tablets, melting at 146°; the latter in long needles, fusing at 132° C. It is very easy to obtain a tangible quantity of the crystals from 50 grammes of the fat. This is saponified with alcoholic potash, the soap well shaken out with ether, the ether distilled off, the residue once more treated with alcoholic potash and ether, and the residue thus purified dissolved on a watch-glass in a few drops of hot alcohol. The long needles of phytosterin obtained in the case of the presence of a vegetable fat can be further tested by the reaction with chloroform and sulphuric acid. Cholesterin gives a beautiful red chloroform solution, phytosterin a bluish one, the difference being strongly marked if the solution is allowed to stand, in a well-corked test-tube, for a couple of days.

I rely, therefore, upon the silver test, coupled with Mauméné's method, and corroborate, if requisite, by that of Salkowski.

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"ON THE ADULTERATION OF LARD WITH COTTON-SEED OIL."

BY ROWLAND WILLIAMS, F.I.C., F.C.S.

(Read at the Meeting, June, 1888.)

DURING the past few months I have had very extensive experience in connection with lard adulteration. Our worthy President, having become aware of the fact, suggested that I should write a short paper, supplementary to the note on the same subject which he himself has read this evening.

It gives me great pleasure to fall in with Mr. Allen's suggestion, and I can only express my regret that my remarks must of necessity be very brief. I should have liked to go into the matter much more exhaustively, but time forbids.

*Saponification equivalent.*—Koettstorfer's process is quite useless, so far as the detection of cotton-seed oil in lard is concerned, as both require practically the same amount of alkali for saponification. This fact will, no doubt, already be so well known to those among you who are regularly engaged in food analysis, that it is, perhaps, almost superfluous to mention it. Determination of the saponification equivalent of lard is, however, occasionally useful, as any great deviation from the normal figure would indicate the presence of some adulterant—cocoanut oil, for instance, which is said to be sometimes used for sophisticating lard.

*Melting Point.*—The melting point of lard is no criterion as to its freedom from adulteration, as the question of a high or low melting point depends entirely upon the part or parts from which the fat is obtained.

*Specific Gravity.*—Inasmuch as the specific gravity of pure lard at 212° Fah. is about .861, while that of cotton-seed at the same temperature is about .872, it seemed possible that the presence or absence of cotton-seed oil might be ascertained by a careful determination of the density of the sample at the temperature of boiling water. I have found that a large proportion of cotton-seed oil does undoubtedly raise the specific gravity of lard to a moderate extent, but a small percentage affects the result very slightly, so that, according to my experience, no very definite information can be obtained by these means.

*Maumené's Test.*—As cotton-seed oil gives a very considerable rise of temperature when treated with strong sulphuric acid, while pure lard gives only a moderate increase, I applied Maumené's test to several suspected samples with a view to ascertain whether cotton-seed oil could be detected in that way, but for some reason or other I did not obtain satisfactory results. Even with samples which undoubtedly contained large proportions of the adulterant, Maumené's test has in my hands failed to determine anything like the real extent of sophistication. I understand, however, that a London chemist has found this method to answer fairly well in the case of lards heavily adulterated with cotton-seed oil, but for my own part I consider Maumené's test too dependent upon mode of manipulation and various other circumstances to yield really reliable information.

*Iodine Absorption.*—This is the test upon which I chiefly rely for the determination of cotton-seed oil in lard, in the absence of interfering bodies. By the latter I more particularly refer to stearin, which is sometimes added in rather large proportion to lard which has been adulterated with cotton-seed oil, in order to neutralise the softening



effect produced by the latter. As stearin absorbs only about one-third as much iodine as cotton-seed oil, the addition of a large percentage of the former naturally reduces the iodine absorption of the mixture to a greater or less extent. Still, I have rarely found the stearin to interfere sufficiently to absolutely nullify the iodine absorption test. Out of the numerous samples of lard submitted to me for analysis, all of those which I believe to be genuine (with one exception) absorbed from 60 to 62 per cent. iodine. Most of these lards, I may say, were of American origin. English lard is said to have a somewhat lower iodine absorption, but I cannot speak with certainty on this point. The peculiar sample just referred to, and said to be leaf lard, absorbed only 51 per cent. iodine—a result for which I am unable to offer any satisfactory explanation.

Mr. Allen informs me, however, that a sample of lard prepared from the omentum, which he has just examined, had an iodine absorption of 55.4 per cent. I myself have to-day rendered some lard from the leaf, and this, I find, absorbs 51.8 per cent. iodine.

I should like to call your attention to the fact that in "Allen's Organic Analysis" (Vol. II., page 50) it will be found that Hübl himself (who may be regarded as the originator of this test) gives the iodine absorption of lard as 59 per cent., whilst other observers give it as 61 per cent., figures so close to my own as to render the probability of 60 per cent. or thereabouts being the true iodine absorption of ordinary lard, made from the fat of the entire animal. I have found samples of refined cotton-seed oil and beef stearin—such as are used for the purpose of adulterating lard—to absorb respectively 110 and 21 per cent. iodine.

Some of the lards which I have examined absorbed as much as 85 per cent. iodine, and I frequently meet with samples having iodine absorptions of 70 to 75 per cent. There can be no doubt whatever about samples such as these being largely adulterated with cotton-seed oil. It should be mentioned that lard dealers can detect even small proportions of cotton-seed oil by the taste and smell, but it has only recently become possible to prove the fraud by scientific evidence.

*Silver Nitrate Test.*—Milliau's modification of Bechi's original process for detecting cotton-seed oil in olive oil has so recently been described in THE ANALYST (May 1888, page 95) that it is unnecessary for me to give the *modus operandi*. I have applied the test to a large number of samples of lard, and find it to answer exceedingly well for the detection of cotton-seed oil. I have examined the fatty acids from numerous other oils and fats, all of which have been free from reducing action on silver nitrate. The reaction seems, therefore, characteristic of cotton-seed oil.

It is advisable to make a blank experiment with the reagents, as I find even "pure" alcohol occasionally contains impurities which reduce silver nitrate to some extent. The silver nitrate test, although undoubtedly of great value as a means of detecting cotton-seed oil in lard, etc., has, I believe, hitherto been used only qualitatively. When employed in the ordinary way it simply indicates the presence or absence of cotton-seed oil, without giving much, if any, idea of the percentage present. During the last few days I have been engaged on a series of experiments with a view to make the process quantitative. So far, however, I have not been successful, but if further experiments prove more satisfactory, I shall be pleased to bring my results before you upon some future occasion.

## LARD ADULTERATED WITH COTTON-SEED OIL.

By E. W. T. JONES, F.I.C.

*(Read at the Meeting, June, 1888.)*

I BELIEVE I was the first public analyst to officially certify under the Sale of Food and Drugs Act this adulteration; hence a few words from me as to the means I rely on for detecting and estimating it may be acceptable to those analysts who may not have yet given the matter their special attention. I do not claim any originality for the reactions involved, but simply point out their application.

The first test I apply is a qualitative one, based on the well-known action of  $S_2Cl_2$  on certain oils. With lard I proceed as follows:—5 grms., which when melted may be pipetted with a tube made to deliver this weight, are put into a small porcelain dish, and just before setting, 2 c.c. of a mixture of equal volumes of  $S_2Cl_2$  and  $CS_2$  carefully pipetted into it, and the mixture well stirred at first, and then occasionally for the first quarter of an hour or so. Do not apply heat, but simply allow to stand on the laboratory table; by this treatment genuine lard only *thickens*, or perhaps becomes *rather stiff*, in three hours; but if it contains any real amount of cotton-seed oil, it becomes quite *hard and solid* in half this time. This test is very simple, but, with practice, one can with a certainty pick out all lards containing cotton-seed oil. For estimating the adulteration I rely on the I absorption from Hübl's reagent, made by dissolving 5 grms. I in 100 c.c. of 95 per cent. alcohol and 6 grms.  $HgCl_2$  in 100 c.c. of such alcohol, then mixing and allowing to stand at least over night before use.

I drop 20 to 22 drops of the melted lard into a weighed 3-oz. wide-mouth stoppered bottle, and carefully re-weigh, the weight of fat not to sensibly exceed 0.5 grm.; just melt by putting the bottle for a minute or so on the top of the water oven, and when nearly cool again, add 10 c.c. of pure  $CHCl_3$ , and so dissolve the fat. When quite cold, add 20 c.c. of the Hübl reagent, very carefully measured, and allow to stand three hours; the colour must then be decidedly brown, showing excess of I, or else *less fat* must be taken for the quantity of Hübl reagent. I object to the addition of more Hübl after the experiment shows too little I, but prefer to commence again with less fat. After standing the prescribed time, the contents of the bottle are transferred to a beaker, the bottle rinsed with KI solution, of which enough must be added to dissolve the I, and the solution then diluted to 150 to 200 c.c., and the free I estimated with  $\frac{N}{10}$  "Hypo" solution and starch; the amount of I now found being taken from what the 20 c.c. Hübl contained leaves the amount absorbed by the quantity of fat employed, and is to be calculated to a percentage.

I standardize the "Hypo" *just before use* with some pure I, carefully weighed (about 0.5 grm.), and so find its *exact* factor.

I take the "Hypo" equivalent of the 20 c.c. Hübl with each set of experiments.

*Example with pure Lard.*

Fat taken, 0.5660 grm. + 20 c.c. Hübl, after 3 hours took 9.4 c.c. "Hypo."

20 c.c. Hübl took 35.6 c.c. "Hypo,"

.5005 grm. I took 39.2 c.c. "Hypo," hence

5005

$39.2 = .0127678$  I for 1 c.c. (Log  $2.1061180$ )

$35.6 - 9.4 = 26.2 \times .0127678 \times \frac{100}{.5660} = 59.10$  per cent. I.



Using logarithms makes the calculation very easy and exact.

I find genuine lard never takes sensibly more than 60 per cent. I, whilst cotton-oil takes from 105 to 110 per cent.

I adopt the formula—

$$100 \left( \frac{\text{I absorbed} - 60}{45} \right) = \text{per cent. cotton-seed oil.}$$

To support the foregoing tests I also take the Sp. Gr. of the lard @ 100° Fahr.

I find the Sp. Gr. of pure lard under such circumstances to be 906·0, and of n-seed oil 913·5, hence—

$$100 \left( \frac{\text{Sp. Gr. found} - 906}{7.5} \right) = \text{per cent. cotton-seed oil.}$$

*Genuine Commercial Lard.*

Sp. Gr. at 100° Fahr.				I per cent.
Not taken	..	..	..	59·90
"	..	..	..	59·10
906·4	..	..	..	56·47
906·4	..	..	..	56·56
906·0	..	..	..	59·04
905·8	..	..	..	57·39
906·1	..	..	..	59·90
906·1	..	..	..	60·42
905·7	..	..	..	59·50
906·0	..	..	..	61·31
Average.. 906·0	..	..	..	58·9

TABLE SHOWING RESULTS CALCULATED FROM THE SP. GR. AND THE I.

	Sp. Gr. at 100° Fahr.	Cotton-seed oil calculated by $100 \left( \frac{\text{Sp. Gr. fd.} - 906}{7.5} \right)$	I <sup>a</sup>	Cotton-seed oil calculated by $100 \left( \frac{\text{I per cent.} - 60}{45} \right)$
pure made of ts lard and art cotton- seed oil.				
{	908·5	33	74·25	32
	908·7	36	76·53	36
	908·4	32	75·26	34
	907·4	18	67·22	16
	906·9	12	65·3	12
	907·5	20	67·3	16
	907·3	17	67·8	17
	907·9	25	69·3	21
	907·4	19	66·7	15
	907·7	23	68·5	19
	907·3	18	68·63	19
	907·2	17	67·7	17
	906·9	13	65·65	13

## DETECTION AND ESTIMATION OF COTTON-SEED OIL IN LARD.

By W. F. K. STOCK, F.C.S., F.I.C.

THE method about to be described is a modification of that devised by Milliau, of which an abstract appears in the ANALYST (vol. xiii., p. 95). As there described, Milliau's process has failed, in the hands of the present writer, to give satisfactory results; but he does not ascribe the irregularities observed to anything but want of detail; for which, probably, Milliau is in no way to blame, since the article in the ANALYST is not an original communication. As worked in the present writer's laboratory the process stands as follows:—

15 grms. of the sample are saponified in a 7-inch porcelain basin with a mixture of 15 c.c. of 30 per cent. NaHO, and 15 c.c. of 92 per cent. alcohol. To commence, the fat is heated to 110°C. The alkaline alcohol must be added in quantities not exceeding 1 c.c. at a time, the temperature not being allowed to fall below 95°C. to 100°C., constant stirring at this part of the operation being most important. If the saponification has been successful, the resultant soap is a smooth thick paste. Boiling distilled water is now added drop by drop, a thin flexible spatula being used to break down the paste. When this has the appearance of smooth starch, water may be run in till a volume of 500 c.c. is reached. Complete solution should follow. 40 c.c. of diluted sulphuric acid (1—10) are now added to the contents of the basin, the liquid is stirred gently and brought to boil for 7 to 12 minutes, then kept just below boiling, until the separated fatty acids fuse to a clear oily layer. The greater bulk of the acid watery liquid is siphoned off, the remainder, with the fatty acids being poured into a clean warm flask with a somewhat long and narrow neck. The fatty acids are freed as nearly as possible by siphonage from the watery under layer, and the flask is filled up with boiling water so as to bring the fatty acids into the neck, by which operation a partial washing is given. 5 c.c. of the fused fatty acids are now transferred by means of a dry, warm, fast-running pipette, into a clean, dry, wide test tube. 20 c.c. of absolute alcohol are added, care being taken to wash the pipette by running the alcohol through it. The contents of the test tube are heated to incipient ebullition in a vessel of boiling water. 2 c.c. of a 30 per cent. solution of silver nitrate are now rapidly poured into the tube, when if even 2 per cent. of cotton-seed oil be present in the sample the characteristic cedar-brown colour is at once developed. Pure lard gives absolutely no colour.

To quantify this reaction, known mixtures of pure lard and refined cotton-seed oil are treated exactly as above, and the colours in the different tubes compared by reflected light against a white background. This must be done simultaneously, for in about seven minutes the colouring matter begins to fall out, and correct comparison is then impossible. In careful hands excellent results are obtainable.

## DISCUSSION.

Mr. HEHNER read the following letter from Prof. J. Campbell Brown:—

University College, Liverpool, June 19th, 1888.

DEAR SIR,—As I cannot attend the meeting of Public Analysts' Society to-morrow, I am anxious that if the paper does not deal with the following points, you



should call the attention of the meeting for me to the two errors analysts are liable to make if they trust to the iodine equivalents already published.

1. They are liable to greatly under-estimate the proportion of cotton-seed oil and other foreign fats in adulterated lard. The substance used by about twenty-five of the American firms is a mixture of cotton-seed oil and beef-stearin, the residue from the manufacturer of oleo-margarine. Now, while the iodine equivalent is high, 105 to 110, the iodine equivalent of commercial stearin is low, under 30. And the iodine equivalent of any mixture that can be used for mixing in large proportions with lard is much lower than that of cotton-seed oil—not higher, if so high, as between 80 and 90. Now if any one calculates the quantity of cotton-seed oil in mixed lard giving an iodine equivalent of say 76, using 105 as the iodine equivalent of the foreign fat, instead of 90 or under, it is clear that he will greatly under-estimate the quantity of foreign fat. He will even under-estimate the cotton-seed oil portion of the foreign fat.

2. They are liable, on the other hand, to condemn genuine lard which is more oily than pork fat or lard rendered in this country.

American lard contains as a rule naturally much more olein than our own. If some of the lard oil has not been pressed out, the high iodine equivalent of lard oil—from 75 to 80—so raises the one equivalent of the thin oily lard, that an analyst judging mainly from the iodine equivalent would infer the presence of cotton-seed oil where there was only excess of lard oil. It is necessary, therefore, to be very careful in determining, first the presence of some cotton-seed oil by safe qualitative tests before determining the iodine equivalent; and it is further necessary, in order to avoid both the errors I have mentioned, to take into consideration the consistency of the sample, and attend to tests for beef-stearin, in drawing any conclusion from its iodine equivalent.

Kindly bring these points before the meeting as from me, unless they are fully gone into in the paper.—I am, yours truly,

The Secretary, Society of Public Analysts.

J. CAMPBELL BROWN.

Mr. HARLAND said that the American manufacturers' plan was to express the lard oil from the lard and replace it by cotton-seed oil. Working on these lines in the laboratory, he had found that about ten per cent. of lard oil can be squeezed out of genuine lards, but that adulterated samples of various American manufacturers gave thirty, forty, or fifty per cent. of what appeared to be, by its sp. gr. and solidifying point, cotton-seed oil.

Mr. WATSON GREY gave the following figures for the percentage of iodine absorbed by various kinds of lard when treated by Hübl's process:—

Kind of lard.	Iodine absorption.
From omentum of hog .....	49.5
Market lard (bought in Liverpool) .....	49
From omentum of sow .....	55.5
From back of pig .....	65

He would fix the average at 57. If this was accepted, it would account for the fact that the President, having taken 62 as the standard number, had found that, whereas the sp. gr. of one adulterated sample corresponded to thirty per cent. of cotton-seed oil, the iodine absorption indicated only fifteen per cent., without having recourse to the supposition that tallow was present as well as cotton-seed oil. His experience went to show that the silver nitrate test—at least, in its unmodified form (without the addition of free nitric acid)—was rather unsatisfactory.

Mr. Fox stated that he had recently found fifty per cent. of earth-nut oil in lard oil, detecting it by the altered sp. gr. and the presence of arachidic acid.

Mr. JONES, in reply to a remark of the President's on his practice of taking the sp. gr. of fats at 100° F., admitted that it was rather an old-fashioned plan, and contemplated changing it. He had not found it necessary to use concentrated aqueous caustic alkali and fresh alcohol when saponifying oils in order to get white fatty acids, as proposed by the President, the alcohol he obtained in Wolverhampton not turning brown from the action of caustic alkali, even when kept for several months.

### "WATER REPORTS—A DILEMMA."

By DR. MATTHEW ADAMS.

(Read at Meeting, July, 1888.)

THE subject of "Polluted Drinking Water and the Closure of Wells," brought forward for discussion by Dr. Hill at the June meeting of our Society, is of sufficient importance to warrant my placing before you a correspondence which passed between me and the Urban Sanitary Authority of Rochester.

This correspondence shows a position of affairs the very reverse of that which Dr. Hill found himself placed in. He fell upon the legal horn; I was impaled by an equally tormenting official horn. In such a dilemma what can be done?

#### WATER ANALYSIS.—REPORT.

To Richard Prall, Esq., Town Clerk.

	No. 1. 61, Morden Terrace, St. Margaret's.	No. 2. 48, Morden Terrace, St. Margaret's.	No. 3. 78, 80, John Street, St. Margaret's.	No. 4. 8, Hooper's Place, St. Margaret's.
Total Solid Matter .....	77·3	57·1	71·4	46·6
Loss on Ignition .....	5·1	3·4	4·6	2·2
Chlorine .....	7·7	5·4	7·0	4·3
Nitrogen, as Nitrates and Nitrites .....	1·14	1·14	1·43	·95
Phosphoric Acid .....				
Free Ammonia .....	·02	·00	·00	·00
Albuminoid Ammonia .....	·05	·04	·02	·02
Oxygen absorbed in 15 minutes .....	·025	·005	·028	·013
Oxygen absorbed in 4 hours ....	·044	·056	·036	·028
Total Hardness (Clark's Scale) ..	27·5	24·0	26·0	20·0
Permanent hardness after boiling ..	12·0	11·1	13·5	9·0
Appearance in 2-foot tube .....	gr. blue	p. blue sus. mat.	pale cl. blue	pale cl. blue
Smell .....	Slight	Slight	Slight	None

☞ All results are given in Grains per gallon, except Free and Albuminoid Ammonia, which are in parts per million.

#### OBSERVATIONS.

County and Borough Analyst's Laboratories,  
Ashford Road, Maidstone.

31st May, 1888.

SIR,

Herewith I hand you results of my analyses of four samples of water recently received from you for analysis, from all of which data I judge that Nos. 1, 2, and 3—especially Nos. 1 and



ore or less tainted, and my advice is they should not be used for drinking purposes if at hand. At the same time I am bound to say there is not much indication of **ACTIVELY** pollution. The polluting matter is for the most part oxidized and so rendered inert, oxidation is not to be always relied upon, therefore such waters must be considered as and dangerous.

I am, Sir,

Yours obediently,

MATTHEW A. ADAMS.

Local Government Board,  
Whitehall, S.W.

September 4th, 1883.

I am directed by the Local Government Board to advert to your letter of the 8th ult., with reference to certain wells in the town of Rochester, that the U.S.A. do not consider could obtain orders to compel the closing of such wells.

Board direct me to state that having regard to the fact that the wells in question supply from a soil which is much befouled by cesspools in near proximity to them, of opinion that measures should at once be taken by the S.A. to secure their closure, an which appears to the Board to be fully borne out by the statements of the Analyst to waters were submitted.

, however, to point out that, quite apart from the positive evidence as to pollution chemical analysis has in this case afforded, the Board in forming an opinion as to the eness or not of a water have regard rather to a full knowledge of its sources and of the ditions by which it is liable to be affected than to its mere chemical ingredients. In etion I am to direct the attention of the Sanitary Authority to pages xvii. to xxi. of anying print of the recent report of their medical officer with especial reference to the xpressed on page xxi., "that we must go beyond the laboratory for evidence of any water being free from dangerous organic pollution."

further to state that the expressions used by the Analyst, and especially with reference luting matter being "for the most part oxidized," appear to show an inadequate view of tions under which water can safely be used.

he present case it appears to the Board that if the Authority are still of opinion that ion might be hindered by some qualifications expressed by the Analyst they should rish analyses of the waters by some well-known chemist, and if necessary the analysis e repeated until any varying qualities of the several well waters have been fully ed, and conclusive inference has been drawn respecting the risk to these sources of

regard to the soakage from cesspools, the Board would call the attention of the Sanitary y to their powers under Section 47 (3) of the Public Health Act, 1875, but they think it add in this connection that to prevent for the future the contents of a privy or cesspool herefrom will not render wholesome the water derived from a soil into which such contents dy soaked in notable quantity.

to add that under Section 62 of the Act, the Sanitary Authority might compel the o, etc., Waterworks Co.'s water to be laid on in any case where it appears on the report rveyor that any house within the district is without a proper supply of water, and that pply of water can be furnished thereto at a cost not exceeding the water rate authorised cal Act in force within the district.

I am, Sir, your obedient servant,

ALFRED D. ADRIAN.

Assistant Secretary.

To Richard Prall, Esq.,

Town Clerk, Rochester.

September 10th, 1883.

R. SIR,

Having regard to my report of May 31st on these four samples of water, and the letter Local Government Board of the 4th inst., I have to remark: that it is not for me as imical adviser "to go beyond the laboratory." It is my duty to form an independent rived from chemical data alone; others, such as your Medical Officer of Health and of Nuisances, must advise you as to local surrounding circumstances; moreover, you ght to expect practical and not theoretical advice, such as may reasonably be expected,

successfully to stand the test of opposition in court; all this you have and no one having respect for his chemical reputation could say more than I have done, viz., that Nos. 1, 2, and 3 are treacherous and dangerous, and should not be used for drinking purposes.

I am, dear Sir,

Yours obediently,

MATTHEW A. ADAMS.

To Richard Prall, Esq.,  
Town Clerk, Rochester.

September 10th, 1883.

DEAR SIR,

I beg to return Local Government Board's letter and printed report of their Medical Officer, together with my remarks thereon. With regard to the "expressions" in my original report referred to in that letter, to the effect that the polluting matter, being "for the most part oxidized," showed (on my part) an inadequate view of the conditions under which water can be safely used, I have simply to say that to the best of my belief, Dr. Dupré, the Local Government Board's own analyst, would endorse every figure and word I have used, and to my mind it would be an undoubted proof of inadequacy and incompetency to lead your authority to overlook the all-important distinction between oxidised and unoxidised organic matter. Their suggestion that the analyses should be repeated until any varying qualities of the several waters have been fully ascertained, etc., is a good one. If on repeated analyses, changes in the constituent parts can be shown to occur, that together with the evidence already attained would put the case in a much stronger light as against the wholesomeness of the waters.

But there is no occasion for this, especially after the Local Government Board's letter; the water ought to be condemned, and if it is necessary I will come over and support this advice in the witness-box.

Yours faithfully,

MATTHEW A. ADAMS.

(*Conclusion of the Society's Proceedings.*)

## ESTIMATION OF GLYCERIN IN THE CRUDE ARTICLE.

R. BENEDIKT AND M. CANTOR.\*

For the determination of the commercial value of crude glycerins, there does not as yet exist an easy and trustworthy process. Most of the processes as yet proposed give uncertain results, particularly the one based on the oxidation with chromic acid, as many other organic bodies are also oxidised to carbonic anhydride and water. The process with permanganate by Benedikt and Zsigmondy has certainly given good results, but in the case of crude glycerin the sample must first be diluted, then treated with subacetate of lead, and the excess of the latter be removed by sulphuric acid before we can titrate.

R. Diez has applied Baumann's test for the estimation of glycerin in wines, etc. The solution is agitated with soda ley and benzoil chloride. The precipitate, consisting of glycerin di and tribenzoate, is weighed, an allowance being made for solubility. Although we will not dispute the use of this process when applied to wine analysis, we have never succeeded in getting correct results with it in the case of crude glycerins, even by scrupulously following Diez's directions. The error amounted from 3 to 8 per cent. of the total glycerin. We now propose the Acetin process, which gives quick and correct results, and is based on the following principle.

Glycerin on boiling with acetic anhydride passes completely into triacetin. If water now be added, and the amount of uncombined acid exactly neutralised with soda, the combined acid may be easily ascertained by saponifying with excess of soda, and titrating back with standard acid. The following solutions are required:—

\* *Zeitschr. f. angew. Chemie*, No. 16.



1. Half normal or normal hydrochloric acid, the exact strength of which must be ascertained with the greatest care.

2. A solution of 20 grammes of sodic hydrate in a litre of water.

3. 10 per cent. caustic soda in a flask holding about  $1\frac{1}{2}$  litre. The flask is closed by an india-rubber cork, through which passes a 25 c.c. pipette, the top of which is closed by an india-rubber tube, and a clamp.

About 1.5 gm. of the sample is weighed in a wide-neck flask with round bottom; 10 grammes of acetic anhydride and 3 grammes of anhydrous sodic acetate are added, and the whole boiled for one and a half hours under an upright condenser. After it is somewhat cooled, 50 c.c. of water are added, and the mixture once more brought up to the boiling point. This must also be done under upright condenser, as the triacetin is sensibly volatile in water vapour.

After the oily mass has dissolved, the liquid is filtered into a wide-necked flask of about 600 c.c. capacity. A white flocculent precipitate, which is rather large in crude glycerins, remains in the filter, and must be well washed. After complete cooling, phenol-phthalein is added, and the fluid *just* neutralised with No. 2 sodic hydrate. The point of neutrality is reached when the fluid turns yellowish-red.

The pipette (No. 3) is now filled, and the contents delivered into the flask. Care must be taken that in doing the check, the pipette shall deliver exactly the same quantity. After boiling for a quarter of an hour, the excess of alkali is titrated back with the acid No. 1.

#### EXAMPLE.

Taken 1.324 grammes of glycerin.

25 c.c. alkali .. .. .	= 60.5 c.c. N acid.
Titrated back with .. .. .	21.5 " "

Triacetin is equivalent to 39.0 " "

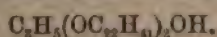
1 c.c. of acid representing .03067 gm. of glycerin, the sample contains 90.3 per cent.

Testing fats for diglycerides: from the ease with which glycerin can be made to triacetin, we may conclude that the mono or diglycerides of the higher fatty acids may be readily converted into triacetin by boiling with acetic anhydride. Another aid to the accurate analysis of fats is thereby obtained, as it is easy to directly estimate the percentage of mono or diglyceride a sample may contain.

Let  $M$  be the molecular weights of the diglyceride,  $a$  the saponification number of the fat *before*, and  $b$  *after* the acetyling, then as 56 stands for KHO, and 42 for the residue  $C_2H_5O$ , the percentage of diglyceride will be—

$$\frac{100 M (b - a)}{5600 - 42 b}.$$

For instance, the stearin from colza oil has to be examined for dierucin



We first determine the saponification equivalent, then *acetylise* 20 to 50 grammes of it, according to the directions of Benedikt and Ulzer, and estimate the saponification

number again. For pure dierucin this is 153.3, for acetylated dierucin 217.4. The molecular weight is 732. If  $a = 46$ , and  $b = 67.8$ , then  $D = \frac{100 \times 732 \times 21.8}{5600 - 42 \times 67.8} = 30$  per cent. dierucin.

If the amount of diglycerides is very small, the combined acetic acid is better estimated by Reichert's process. The calculation is somewhat more troublesome if the fat contains glycerides of oxyacids, as these give also an acetyl number. In this case we isolate the fatty acids first, and then determine their acetyl equivalent.

Reimer and Will particularly point out that colza oil contains an oleic acid, which is isomeric with the oleic acid of castor oil. This is, however, contradicted by its low acetyl number, which they found to be 6.3, corresponding with 3.8 per cent. oxyoleic acids. The true composition of colza oil is not as yet known with certainty, then its diene absorption power 100, whilst the acids which Reimer and Will have isolated give a far lower figure: erucic acid 75.2, benic acid = 0, rapinic acid 85.2. It is, therefore, probable that a large proportion of another acid of the linseed oil type must be present.

Dierucin is the only glyceride as yet isolated from natural fats, but it must yet be ascertained if it really exists in the fresh oil.

Allen has lately suggested that Japan wax contains dipalmitin. The direct test gave a negative result. The saponification equivalents of the original and acetylated wax were found to be the same, viz., 222.

On saponifying butter with an insufficiency of alcoholic potash, Bell got an oily substance, melting at  $4.4^{\circ}\text{C}$ . We have repeated this experiment, and indeed obtained a thinnish oil, with a strong butyric ether smell, which, however, did not yield any glycerin, and must therefore have consisted for the greater part of ethylic ethers.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

AVOIDING THE USE OF WEIGHED FILTERS AND THE REDUCTION OF PRECIPITATES BY THE FILTER. LL. DE KONINCK. *Zeitschr f. anal. Chemie*, No. 15.—About twelve years ago, a method for the estimation of potash was recommended by Fresenius, whereby the use of the weighed filter was avoided. The platinum precipitate was collected on a filter, and after drying, the bulk of it was carefully removed. The minute quantity still adhering was now removed by treatment with boiling water, and this solution evaporated in a weighed dish. After adding the chief precipitate, the whole was dried and then weighed. The author has extended the idea to a number of other metals.

AMMONIUM.—The platinic ammonium precipitate may be treated like the potassium compound.

ARSENIC.—The triple arseniate is carefully removed from the filter and put into a crucible. The filter is repeatedly washed with small quantities of dilute nitric acid, and the liquid is evaporated to dryness in the same crucible. When completely dry the whole is gradually heated to redness, when a pure magnesian pyro-arsenate is formed without the least loss of arsenic.



**MAGNESIA OR PHOSPHORIC ACID.**—The triple phosphate obtained is treated in similar manner. A pure white pyro-phosphate is obtained instead of the blackish-looking ones usually got.

**ZINC AND CADMIUM.**—When the filters containing their carbonates are burnt, an appreciable loss is sustained, particularly in the case of cadmium. The best way is to remove bulk of the precipitate, and to dissolve out remainder of zinc or cadmium with dilute nitric acid, which solution is then evaporated to dryness in a weighed dish. The dried precipitate is now added, and the whole ignited and weighed.

*Note by abstractor.*—The idea of dissolving a precipitate of the filter by means of nitric acid has already been applied in the case of zinc by Dr. S. Bein. See ANALYST, N. xi., p. 135. L. DE K.

**ON THE EMPLOYMENT OF DIAZO COMPOUNDS FOR THE DETECTION OF ORGANIC IMPURITY IN WATER.**—In a recent number of the *Berichte der Deutschen Chemischen Gesellschaft* (xxi., No. 9), Hr. Griess advocates the use of paradiazo-benzol-sulphuric acid for the detection of organic impurity in water. It has been known for some time that an alkaline solution of the diazo-compound gave a yellow or orange colour with human urine; further research now shows that by this colour reaction, 1 part of human urine in 5,000 of water, and 1 part of horse urine in 50,000 parts of water can be accurately and certainly detected and estimated. 100 c.c. water mixed with  $\frac{1}{10}$  c.c. human urine gave, with a few drops of the diazo solution, a colour about equal to that produced by  $\frac{1}{20}$  mgr.  $\text{NH}_3$  in 100 c.c. water with Nezsler's reagent. The diazo solution is made by dissolving 1 part of para-diazo-benzol-sulphuric acid in 100 parts of water, and adding NaOH solution until slightly alkaline. This solution must be used while quite fresh, as it turns yellow on keeping. The method of applying the test is very simple: to 100 c.c. water a few drops of the diazo solution are added, well mixed, and the colour produced observed. If after standing five minutes no colour forms, the water can be considered pure. It may be remarked that the colour produced by cane sugar in solution is very faint, and that diabetical urine containing 6 per cent. cane sugar gives much less colouration than normal urine. J. B. T.

#### LAW NOTES. TO OUR READERS.

Some years ago it was decided to discontinue the reporting of ordinary police-court proceedings, and to give such cases as authoritatively established some point in connection with the working of the Acts which public analysts are interested. After a fair trial of this system, a majority of the members of the Society have expressed a wish that our old practice should be, to some extent, returned to, and, in deference to such request, we have decided to resume the reporting of police proceedings. The line will be drawn at accounts of ordinary cases, and reports will only be inserted when any novel, legal, or chemical question arises, or where the certificate of the analyst is in any way attacked. Any member or subscriber desiring to have such a case, therefore, invited to furnish us with a report of the proceedings, signed with his name, not for publication, but as a guarantee of exactitude.

**THE ADULTERATION OF AMERICAN LARD.**—Before the Potteries Stipendiary (Mr. H. C. Greenwood), at Longton, July 4th, there was an investigation of several cases involving the alleged adulteration of lard imported from America. The cases were brought before the Court by Mr. E. W. H. Wright, inspector under the Adulteration of Food and Drugs Act; and Mr. F. Smith, of Liverpool, appeared for the defendants, and likewise watched the proceedings on behalf of the wholesale dealers. In the first case, John Siddall, grocer and provision dealer, Dresden, was charged with having sold lard that was certified by the County Analyst to be adulterated to the extent of 15 per cent. with cotton-seed oil. The case was before the Court, May 30th, when it was stated that it was one of the first of the kind in the country, cotton-seed oil never having been alleged as an adulteration before. At the request of Mr. Smith, a sample of the lard was ordered to be sent to the Government



laboratory at Somerset House, and the case was adjourned to await the result. It was now stated that the Government Analyst certified that the lard contained 20 per cent. of cotton-seed oil.—Mr. Knight asked that an exemplary penalty might be imposed, as, he said, it was very evident that it was not the retailers, but the wholesale dealers who were the offenders, and were defending the matter. In support of this statement, he pointed out that the wholesale dealers had issued a circular to the retailers stating that if anything was wrong with the lard they would make it all right. This, he contended, was practically saying that they would pay any fine that might be inflicted.—Mr. Smith said that no one was more surprised than the wholesale dealers when this American lard was found to be adulterated, for until Mr. Jones (the county analyst) detected that it contained cotton-seed oil there was not an analyst in the country who could say that it was not pure. As a matter of fact some analysts had certified that it was pure. The wholesale dealers were innocent of the adulteration, and they therefore stood at the back, in a reasonable and proper way, of those persons whom they had innocently misled.—The Stipendiary said that he had no doubt that the adulteration was done in America, and that the defendant knew nothing about anything having been added to the lard, but, unfortunately for him, the law held him responsible. As this was one of the first cases of the kind, he should treat it as a first case, but now that it was becoming known that the lard was adulterated he should severely punish any future offenders. The defendant was fined 40s. and 19s. costs.—The following tradesmen were also charged with having sold lard adulterated with cotton-seed oil: Samuel Jenkinson, grocer, High-street, Longton, 20 per cent.; Henry Dayson, grocer, Market-street, 17 per cent.; and Thomas Pickering, grocer, High-street, 19 per cent. The certificates of the analysts were not disputed, and the magistrate imposed a fine of 40s. and 19s. costs in each case.

**"PURE AND REFINED LARD" FROM AMERICA.**—**IMPORTANT PROSECUTIONS.**—At the Wednesday Police Court, July 3rd, before the Stipendiary (Mr. Neville), George Blackham, wholesale grocer, Darlaston, was charged with selling lard not of the nature and quality demanded.—Mr. R. A. Willcock (Wolverhampton) appeared to prosecute on behalf of Mr. J. E. Morris, the Inspector under the Food and Drugs Act; and Mr. James Slater defended.—In opening the case, Mr. Willcock stated that on June 1st Mr. Morris visited a shop at Darlaston occupied by the defendant, and there saw exposed for sale two piles of lard marked respectively 5d. and 6d. per lb. Mr. Morris asked for a pound of lard at 5d., with which Mr. Blackham supplied him, and for which Mr. Morris paid. A conversation ensued, in the course of which Mr. Blackham said he believed it was pure lard, and if it were not it was very hard that the retailer should be prosecuted.—Mr. Morris said he quite agreed with him, and a bucket was then produced, on the lid of which was printed "Armour and Co., pure and refined lard, Chicago, U.S."—The lard was subsequently analysed, and was found to contain 17 per cent. of cotton-seed oil. This was first discovered by Mr. E. W. T. Jones, and the wholesale house had now recognised their liability, and had sent out a circular to their customers in these terms:—"Armour's Refined Lard.—It has been decided that Armour's refined lard, as hitherto imported from Chicago, cannot be sold as pure lard, but as mixed. We shall be happy to make any reasonable allowance that you may think needful to repay you for any temporary trouble or inconvenience you may experience in selling the mixed as distinct from the pure lard, or take back any unsold portion at your option later on should you find any trouble, which we hardly anticipate, as Messrs. Armour's refined lard is of unimpeachable quality for domestic purposes—(laughter)—and contains the most thoroughly health-giving properties, and is extensively used by the general public all the world over.—Pelling, Stanley, and Company, Liverpool, June 20, 1888." Together with this was a copy of label which should be supplied to every purchaser. It was "Armour's refined lard. This product is mixed."—Mr. Morris having given evidence in proof of this statement, Mr. Slater for the defence said defendant purchased the lard in question as pure and refined, from Messrs. Carter and Wooding, of Liverpool, who received it from Pelling, Stanley, and Co., the agents for Messrs. Armour. To show what the profit was like, he might mention that defendant paid 42s., including carriage, for a hundredweight of lard, which was equal to 4½d. per pound. He sold it at 5d. per pound, and thus only realised a profit of ½d. per pound.—The Stipendiary said he did not understand the prosecution to suggest that the English retail dealers got large profits. It was the Americans who were said to be doing that.—Mr. Slater then went on to contend that Mr. Blackham received a written guarantee within the meaning of the Act that the lard was pure. This, he submitted, was established by the fact that the lard-bucket lids had printed on them "Armour and Co., pure and refined lard, Chicago."—Mr. Slater argued this point at length, but the Stipendiary said he decided that that was not a written warranty sufficient under the 25th section of the Act. So far as defendant was concerned he no doubt bought the lard as pure, and had not made much profit out of it. The profit was unfortunately got across the water.—Defendant would be fined £5 and costs, for a penalty of that kind seemed to be the only way to stop the traffic. He had not the slightest doubt defendant would be able to recover the fine. Wm. Wilkes, grocer, Darlaston, was charged with a similar offence, the lard in this case being adulterated with 19 per cent. of cotton-seed oil.—Mr. Frederick Smith, of Liverpool, solicitor to Messrs. Pelling and Stanley, appeared for the defence, and urged that a nominal penalty would meet the case.—His Worship imposed a fine of £5 and costs, remarking that Mr. Jones had acted the part of a public benefactor by discovering that adulteration. A good deal of profit must have been made somewhere.—Mr. Smith: It is in America.—Walter Raybold, Dudley Port, grocer, charged with a similar offence, was fined £5 and costs.—In the first case Mr. Slater gave notice of appeal, and the Stipendiary expressed his willingness to grant him a case.—The amount of fine and costs in each of the cases was £6 16s. 0d.



# THE ANALYST.

OCTOBER, 1888.

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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

### THE USE OF THE TERM "NORMAL" IN VOLUMETRIC ANALYSIS.

By ALFRED H. ALLEN, F.C.S., F.I.C.

(Read at Meeting, July, 1888.)

THERE is one point which has attracted my attention a good deal from time to time, and which appears to me a very proper subject for the Society of Public Analysts to consider, and even to express a formal opinion on. I refer to the definition of normal solutions. Unfortunately, the term "normal," as applied to volumetric solutions, is used in two distinctly different senses. By Mohr, whom we may regard as the father of volumetric analysis, and Sutton, whose book is best known to English chemists, the term is employed to signify a standard solution containing one equivalent of the active constituent in 1000 measures of water. The exact definition of a normal solution used by Sutton is that one litre at 16°C shall contain the hydrogen equivalent of the active reagent weighed in grams. ( $H=1$ ). In Vol. I. of my *Commercial Organic Analysis*, I have practically adopted the same definition, describing a normal solution as "one containing in 1000 c.c. such an amount of the active constituent as will combine with, replace, or oxidise one gramme of hydrogen." Tollens similarly defines normal solutions as "those containing in one litre that quantity in grammes of the active substance which, in their action under consideration, is equivalent to one molecule, or 36.5 grammes of hydrochloric acid." In the above definitions all normal solutions are of exactly corresponding strength, and those of a similar nature may be substituted one for another, volume for volume.

Thus, according to this definition, which may be called the "equivalent system," the following are the strengths of typical normal solutions:—

			Grms. per litre.
Normal caustic soda	contains	Na	=23
" " "	"	NaHO	=40
" sodium carbonate	"	Na	=23
" " "	"	Na <sub>2</sub> CO <sub>3</sub>	=53
" " "	"	2	=53
" hydrochloric acid	"	HCl	=36.5
" sulphuric acid	"	H <sub>2</sub> SO <sub>4</sub>	=49
" " "	"	2	=49
" oxalic acid	"	H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> , 2 H <sub>2</sub> O	=63
" " "	"	2	=63

Similarly, the following solutions are decinormal:—

			Grms. per litre.
Decinormal silver nitrate	contains	AgNO <sub>3</sub>	=17.0
" mercuric chloride	"	HgCl <sub>2</sub>	=15.55
" potassium permanganate	"	KMnO <sub>4</sub>	= 3.162
" iodine	"	I	=12.7
" sodium thiosulphate	"	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> + 5 H <sub>2</sub> O	=24.8
" " "	"	10	=24.8
" arsenious acid	"	As <sub>2</sub> O <sub>3</sub>	= 4.95
" " "	"	4	= 4.95

By the other school, a normal solution is understood to mean a liquid containing the *molecular weight* in grammes in 1,000 c.c. By Fleischer, one of the advocates of this system, the old atomic weights are used, so that in most cases the strengths of his solution are the same as those made on the equivalent system, but in the translation of Fleischer's book, by Mr. M. M. Pattison Muir, the modern atomic weights are used, with the consequence of very serious obscurity. Thus while normal caustic potash would contain 39.1 grammes of K per litre, we are told that "a normal solution of potassium carbonate is prepared by dissolving 138.2 grammes of potassium carbonate in 1,000 c.c. of distilled water. Hence such a solution contains 78.2 of K, and is twice as strong as the caustic alkali, or as the normal potassium carbonate of the original German.

The confusion is still greater when permanganate comes to be used. In the original German, Fleischer calls a solution of 32 grammes of potassium permanganate in 1 litre  $\frac{2}{10}$  normal. It is true that this solution is absurdly strong, and is capable of doing ten times the work attributed to it by the author. Mr. Muir has corrected this in an *erratum*, but in the text of his translation he actually doubles the strength of the above solution, recommending one containing 64 grammes per litre.

That the nomenclature of a standard solution should vary according as the writer employs the old or doubled atomic weights must be held to be highly inconvenient, to say the least of it, though Sutton (compare first with later editions), by employing the "*equivalent system*," has wholly avoided any such source of confusion. According to the



principle adopted by the "molecular weight" advocates, the employment of the same atomic weights will not always save us from confusion, for a standard solution of permanganate will be differently called according as its employer considers the salt in solution to be  $\text{KMnO}_4$ , or disregards its isomorphism with  $\text{KClO}_4$ , and prefers to write it  $\text{K}_2\text{Mn}_2\text{O}_8$ .

Professor Dittmar, in his recently published work on "Chemical Analysis," wholly avoids the term normal, as he does also in his article on the subject in the new edition of "Watts' Dictionary of Chemical."

Dr. Attfield also avoids the use of the term "normal" both in his *Chemistry*, and in the *British Pharmacopæia*, preferring to adopt round-about and not always very intelligible phrases in its place.

In a paper by C. Winkler (*Berichte*, xviii., 2,527), a short abstract of which appears in the *Journal of the Chemical Society*, vol. I., page 96, the author contends that the volumetric system should be derived from the molecular weights, and not from the equivalent weights as is the case at present. Tollens deprecates the proposed change (*Jour. Chem. Soc.*, I. 1,070) on the ground that it would cause confusion, and suggests the definition of a normal solution already quoted.

Mr. John Pattinson evidently prefers the "equivalent" application of the term normal (*Jour. Soc. Chem. Ind.*, vi., 351.)

The most recent support of the "molecule" content of normal solutions is that given by our esteemed ex-president, Dr. Muter, whose experience as a practical analyst and a teacher gives his opinion great weight. On page 105 of the new edition (3rd) of his *Manual of Analytical Chemistry* he defines a normal solution as one "having one molecular weight in grammes per litre." The subsequent pages afford some striking illustrations of the inconsistencies into which the supporters of this system are unavoidably driven. Thus every one knows that the molecule of iodine is  $\text{I}_2$  and has the weight 254, yet  $\frac{\text{N}}{10}$  solution of iodine is directed by Dr. Muter to be prepared with 12.7 grammes per litre, instead of 25.4 grammes.

It would be interesting to know what strength the supporters of the "molecule" definition would attribute to decinormal solution of arsenious acid. The solid substance, or white arsenic, according to many authorities has the molecular constitution  $\text{As}_4\text{O}_6$ , though it is more commonly looked on as  $\text{As}_2\text{O}_3$ . But the solution admittedly contains  $\text{H}_3\text{AsO}_3$ , or when neutralised  $\text{Na AsO}_2$ . Query: Is a solution containing one, two, or four atoms of As in decigrammes per litre to be regarded as decinormal? By the "equivalent" system of definition there is no difficulty, for a decinormal solution will be one which will react with an equal volume of decinormal iodine.

According to the supporters of the "molecule" system, decinormal bichromate solution will contain 29.5 grammes of  $\text{K Cr}_2\text{O}_7$  per litre. This will oxidise  $\frac{\text{H}_2}{10}$  and hence be six times the strength of the solution similarly named on the equivalent system. But if used to precipitate lead or barium it will replace  $\frac{\text{H}_2}{10}$ . Here is an instance of confusion in the case of the "equivalent" nomenclature, I believe the only one.

(Conclusion of Society's Proceedings.)

## QUANTITATIVE ESTIMATION OF PARAFFIN, CEROSIN, AND MINERAL OILS IN FATS AND WAX.

By F. M. HORN.\*

THE processes devised by Geissler give inaccurate results. The proposal of Allen and Thomson to treat the dried soap with petroleum ether (volatile at  $80^{\circ}\text{C}.$ ) gives, according to Benedikt, useful results in the estimation of the unsaponifiable principles of pure animal and vegetable oils, but is not to be trusted if these contain mineral oils, because their soaps are partly soluble in petroleum ether. Instead of using petroleum ether, it has been tried to treat the soap in a Soxhlet's apparatus, with boiling chloroform, in order to extract the paraffin. The test analyses were very satisfactory, and gave results within .2 per cent. To insure rapid and complete saponification the following plan will be found advantageous:—About 6 grammes of the sample to be tested is put in a porcelain dish, a piece of caustic soda weighing about 3 grammes is added, and after adding 80 c.c. of alcohol, the whole is heated on a waterbath, and well stirred with a glass rod. The saponification goes on very quickly, and it does not take long before the soap is quite dry. Addition of sand or sodium carbonate is superfluous. The excess of alkali is gradually made into carbonate by the carbonic acid of the atmosphere. When dry, the soap is put into a filter-paper cartridge. The basin may be rinsed with a little chloroform, which is then poured into the mass after it has been put into the Soxhlet. The cartridge must be closed at the bottom, but not on the top. The extraction by means of chloroform now goes on as usual; and if the liquid should be turbid, it must be filtered. After evaporating the chloroform in a weighed dish, and drying residue at  $110^{\circ}\text{C}.$ , the residue may be weighed. The quantity of paraffin in composite candles may be very accurately determined by this process.

In applying the process to wax, it must be remembered that beeswax contains about 50 per cent. of unsaponifiable matter (myricylic alcohol), which is soluble in chloroform. This must, of course, be separated from the paraffin. 6 grammes of the suspected wax are saponified as described, and extracted. The residue from the evaporation of the chloroform is boiled with acetic anhydride, when everything fuses. The myricylic alcohol is, however, gradually converted into an acetate, and dissolves; whilst the paraffin collects in drops on the surface of the fluid. The mixture must now be filtered through a thick filter (the funnel to be surrounded by hot water), and the paraffin thoroughly washed with acetic anhydride. When sufficiently washed, the acetic acid is removed by washing with boiling water. No loss in paraffin is to be feared as long as the filter contains some water; but if allowed to run dry, paraffin may go through the paper. When all acid has been removed, the filter is put into a small beaker, dried at  $100^{\circ}\text{C}.$ , and the paraffin extracted by petroleum ether or chloroform. After evaporating off the solvent, the residue is dried and weighed. Instead of using acetic anhydride, glacial acetic acid may be used; but as the myricylic acetate is then far less soluble, it is more difficult to wash out.

\* *Zeitschr. f. angew. Chemie*, No. 16.



The process succeeds equally well in presence of rosin, which is almost invariably present with the paraffin.

## TEST ANALYSES.

Mixture consisting of—				Found.	
Tallow	and 22.7 per cent. paraffin	...	...	22.6	per cent. paraffin
Stearic acid	and 9.58 "	"	"	9.75	" "
Castor oil	and 20.6 "	"	vaseline oil	20.4	" vaseline oil
Cotton oil	and 14.23 "	"	mineral oil	14.4	" mineral oil
Beeswax	and 17.42 "	"	paraffin	17.88	" paraffin
White wax	and 19.20 "	"	"	19.1	" "
Wax, rosin, and	14.0 "	"	"	14.2	" "

### NEW METHOD FOR THE DETERMINATION OF CARBONIC ACID IN THE AIR.

BY G. LUNGE AND A. ZECKENDORF.\*

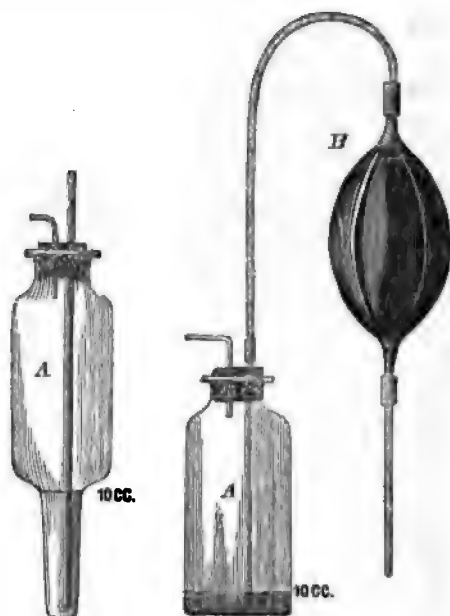
It is a well-known fact that a rapid estimation of the carbonic acid in the air, without complicated apparatus, is still a desideratum from a hygienic point of view. A process devised by R. Angus Smith, slightly modified by one of us, was published in German in 1877. The minimetric apparatus then described was based on the fact that the purer the air the more volume of it is required to cause a decided turbidity with lime or baryta water. The difficulty is, however, to notice the exact moment turbidity sets in, and therefore one of us searched in vain for an indicator to show the exact point. This was in 1876 and 1877.

Since that time many chemists have proposed the use of phenol-phthalein. Blochmann, Balló, Schaffer, and Wolpert have devised apparatus, which are, however, either far too complicated for the use of sanitary inspectors (without being, after all, very accurate) or very simple but very imperfect. Some of them, having been patented, are also rather expensive. The consequence is that officials who ought to use them, as a rule, do not trouble about the matter. As will be seen, one of us has succeeded in perfecting the popular minimetric apparatus, but we will first review some other processes.

Blochmann makes use of a half-litre flask, into which small volumes of lime-water containing phenol-phthalein are successively poured, until the red colour no longer disappears. (One volume of the lime-water absorbs the normal quantity of the carbonic acid.) Balló also uses a half-litre flask connected with a burette, from which are delivered small portions of potash-lye, coloured with phenol-phthalein and containing some barium chloride. After every addition the flask must be well shaken, until the liquid is decolorised. Both authors then simply report, Carbonic acid normal, or about so much in excess. Schaffer and Wolpert moisten a piece of blotting-paper with a solution of sodium carbonate, coloured with phenol-phthalein, which will be gradually decolorised by the carbonic acid in the air, the carbonate passing into bi-carbonate. The more carbonic acid the air contains, the quicker the paper will lose its colour. The process, though simple, is not trustworthy, as it depends on too many circumstances, such as currents of air, and therefore may lead to great mistakes. Far better and even

\* *Zeitschr. f. Angew. Chemie*, No. 14.

comparatively accurate results are obtained by the use of phenol-phthalein in the minimetric apparatus. It is, however, plain that the apparatus in its old form is not well adapted for the purpose. With the old indiarubber ball of about 22 c.c. capacity, one must either work a very long time or use enormously diluted solutions, which may give rise to serious errors, as diluted solutions so soon decompose, and the change of colour then also becomes uncertain. Therefore we had to use a larger indiarubber pump. It has, further, been almost unavoidable to provide the pumps with valves, as they are used for medicinal purposes. Experiments carried out by Mr. Bertschinger proved *those* valves to be the best which were constructed on the system of the heart. We now use an indiarubber pump provided with such valves, which may be still easily compressed



with the hand, and still delivers a fairly constant volume of air, say from 70, 68, 72, 68, 71, 72, to 71.5 c.c., therefore an average of 70.3 c.c., with an error of 1.7 c.c., which would only influence the fourth decimal of the percentage of carbonic acid. We have also made a mechanical pressing apparatus, by which the error may be reduced to .5 c.c., but this arrangement will only make the apparatus less suitable for those persons for whom it is really intended, without giving a corresponding advantage. The same may be said of the substitution of the indiarubber ball for a glass or metallic pump in the shape of a stomach-pump.

The larger capacity of the new indiarubber pump, as compared with the old one, also rendered necessary the use of a larger flask. One having a capacity of 110 c.c., 10 c.c. for the reagent, and 100 c.c. for the air, proved satisfactory. We found it better not to draw the air through the apparatus, but to force it through.

The illustration shows the apparatus (one quarter of its natural size). Flask A has a ring mark, showing 10 c.c., which volume is, however, much better measured with a

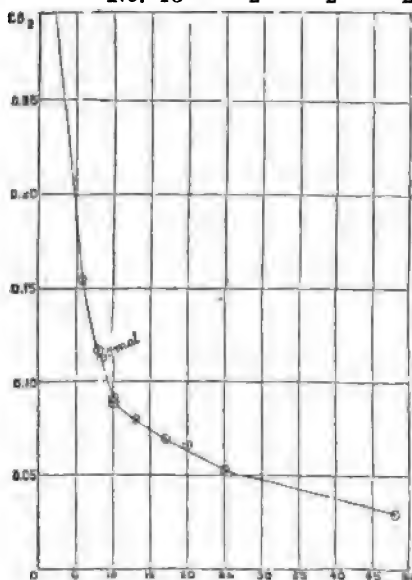


pipette. Or the flask may be drawn out, which admits of a more accurate measuring of the fluid and a more thorough passage of the air, but its shape makes it, again, less practical. When the apparatus is required for use first press the indiarubber ball (B) with the right hand, and repeat this a few times, that it may be filled with the air under examination. The flask is now opened, and 10 c.c. of the reagent are now introduced. After closing, the contents of B are now slowly pressed into A, which must be well shaken, more particularly towards the end. The red reagent, consisting of  $\frac{N}{500}$  solution of sodium carbonate, containing .02 gramme of phenol-phthalein per litre, gradually gets paler. If the air is very impure a couple of volumes *b* will completely decolourise it. In the case of moderately impure air, from 9-10 volumes will be required, for ordinary air of the town about 25 volumes, whilst for pure country air, about 40 volumes will be required. In the latter case, a complete decolorising seldom succeeds, and one must cease transmitting more air, when the colour does not seem to fade any more. As, however, the testing of *pure* air is out of the question, this uncertainty of the final reaction does not affect the process, and an excess of 3 volumes only affects the percentage about .005 per cent.

The strength of the reagent as mentioned has proved itself to be the best. If stronger, a very large volume of air must be passed through; if weak, it too soon decomposes. It is therefore better to use a  $\frac{N}{10}$  solution, made of 5.3 grammes dry sodium carbonate in one litre of water, with the addition of 1 gramme of phenol-phthalein dissolved in alcohol (to be added before the whole is made up to 1 litre) or this may be added in powder, and dissolved by gentle warming. Before use, 2 c.c. of this solution are diluted with water (free from  $\text{CO}_2$ ) up to 100 c.c., and 10 c.c. of this fluid used for each experiment. This solution keeps a long time, but if it is some weeks or even days old it is safer to make it afresh. Long experience has taught us that the kind of glass is without influence, the solution keeping as well in Thüringen as in Bohemian glass bottles. As will be easily understood, the air previously contained in the flask A also takes part in the reaction; but as this quantity is constant, no correction was required in making up the table (see the end of this article).

It must not, however, be imagined that the amount of  $\text{CO}_2$  of the air may be calculated from theorising that the sodium carbonate passes completely into bicarbonate. Percentages thus calculated are far from what they really are, and the more inaccurate the poorer the air is in carbonic acid. This is only quite natural, then it is not possible to combine the last trace of sodium carbonate with the  $\text{CO}_2$  even after prolonged shaking; in fact, solution of sodium hydro-carbonate when shaken with air free from  $\text{CO}_2$  actually parts with traces of this gas. The relation between the number of fillings and the amount of carbonic acid had, therefore, to be ascertained by direct experiment. A small room, used for no other purposes, and to which there was no admission for strangers, was filled with air mixed with a definite amount of carbonic acid. The air was thoroughly mixed by means of moving a large cardboard, and the  $\text{CO}_2$  estimated twice with the minimetric apparatus. Six litres of the air were then collected for the purpose of later on estimating the  $\text{CO}_2$  by Pettenkofer's process, and then two more minimetric estimations were performed. Of the following experiments, the first one was not quite satisfactory, through want of practice and insufficient mixing of the air.

Number of Volumes of Air with the Minimetric Apparatus.						Carbonic Acid by Pettenkofer's Process	
No.	1	48	49	48		Average.	(parts in 10,000).
No. 1	48	49	48			48	3.01*
No. 2	25	25	24			25	5.32†
No. 3	26	25	26	26		26	5.96†
No. 4	20	21	20	20		20	6.62
No. 5	17	17	18	17		17	6.91
No. 6	13	13	12			13	8.06
No. 7	10	9	10	10	9	10	8.90
No. 8	10	11	10	10	11	10	9.15
No. 9	8	8	9	9		8.5	11.28
No. 10	8	8	8			8	11.70
No. 11	8	8				8	11.70
No. 12	6	6	6			6	15.55
No. 13	2	2	2			2	30.0



From the graphic illustration it will be noticed the results may be expressed by a well-defined curve, the only deviation being No. 3; but as we have already explained, with a pure air like this the end reaction is not so easily noticed and the error but very small. One may, therefore, construct by graphic interpolation a table which gives for a given number of fillings the quantity of carbonic acid. This table is, of course, only suited for an apparatus of the same size as we have used, which may be obtained for 7s. 6d. from J. G. Cramer, in Zürich. For different-sized apparatus, other tables must be constructed.

It must be further mentioned that our experiments were done at a temperature of about 18° C, which, however, will be the average temperature of most localities to be tested, so no correction will be required. The barometric pressure was about 730 mm. Great differences in pressure in other localities may necessitate a slight correction, but for all practical purposes may be safely disregarded.

Table showing the Percentage of Carbonic Acid (Pettenkofer) Corresponding with every Filling of the Ball of the Minimetric Apparatus.

No. of Fillings:	CO <sub>2</sub> per cent.	No. of Fillings.	CO <sub>2</sub> per cent.
2 .. .. .	.30	16 .. .. .	.071
3 .. .. .	.25	17 .. .. .	.069
4 .. .. .	.21	18 .. .. .	.066
5 .. .. .	.18	19 .. .. .	.064
6 .. .. .	.155	20 .. .. .	.062
7 .. .. .	.135	22 .. .. .	.058
8 .. .. .	.115	24 .. .. .	.054
9 .. .. .	.100	26 .. .. .	.051
10 .. .. .	.09	28 .. .. .	.049
11 .. .. .	.087	30 .. .. .	.048
12 .. .. .	.083	35 .. .. .	.042
13 .. .. .	.08	40 .. .. .	.038
14 .. .. .	.077	48 .. .. .	.030
15 .. .. .	.074		

\* Air from Zurichberg.

† Air from the room without added CO<sub>2</sub>.



In conclusion, we must not omit to mention that we have also tried the use of alcoholic solutions. As is well known, alcoholic alkaline solutions, coloured with phenolphthalein, soon fade, which, according to Draper, is caused by the absorption of  $\text{CO}_2$  from the air, which not only dissolves quicker in alcohol than in water, but also precipitates alkaline carbonate. Decolorisation, therefore, takes place long before the bicarbonate is formed. Our experiments have, however, shown that this reaction is useless to us, on account of its too great delicacy. Alcohol kept boiling for hours, and allowed to cool, with complete exclusion of  $\text{CO}_2$ , still decolorised  $\frac{N}{10}$  solution. It therefore either retains  $\text{CO}_2$  or its decolourising power must be caused by some other impurity.

### ADULTERATION OF LARD WITH COCOA-NUT OIL.

By ALFRED H. ALLEN.

I AM induced to make this communication, without waiting for a meeting of the Society of Public Analysts, on account of the very active interest now attaching to lard adulteration. In the last edition of my "Commercial Organic Analysis" (Vol. ii., page 142) the assertion is made that "cocoa-nut oil has been employed for adulterating lard," but I am unable to trace the authority on which I made the statement. Personally, I never found, nor, indeed, looked for, cocoa-nut oil in lard until quite recently, as I have regarded the peculiar odour or flavour of cocoa-nut oil as an inseparable barrier to its unacknowledged use. This difficulty is now surmounted. A few weeks since I received a sample of lard for examination under the Sale of Food and Drugs Act, which, on analysis, gave the following results:—

Water .. .. .	0.86 per cent.
Indicated plummet gravity at $99^\circ \text{C}$ . ..	.8666
Iodine absorption .. .. .	37.4 per cent.
Nitrate of silver test .. .. .	negative.

These results were so extraordinary that I at once suspected the presence of cocoa-nut oil, and this suspicion was fully confirmed by the following additional data:—

#### ON ORIGINAL FAT:—

KHO required for saponification .. .. .	21.15 per cent.
= Saponification equivalent .. .. .	265.2
Volume of $\frac{N}{10}$ alkali required by the distillate from 2.5 grms. by the Reichert-Wollny process ..	3.3 c.c.

#### ON SEPARATED FATTY ACIDS:—

Mean combining weight .. .. .	253.04
Volume of $\frac{N}{10}$ alkali required by distillate from 5 grms. ..	3.5 c.c.
Plummet gravity at $99^\circ$ .. .. .	.8400
Iodine absorption .. .. .	42.5 per cent.

The volatile acids obtained by the Reichert-Wollny process contained a notable proportion of solid acids of sparing solubility in water, and had the characteristic odour of the distillate from cocoa-nut oil. I certified the sample to contain 33 per cent. of the adulterant.

It is evident that the very characters which render it difficult to detect and determine cocoa-nut oil in butter suffice to make its detection and determination in lard, even in presence of cotton-seed oil and tallow, a certain and fairly simple matter. This will be evident from an inspection of the following figures:—

ORIGINAL FAT:—	Lard.	Cocoa-nut Oil.
Plummet gravity at 99° C. ..	860 to 861.	868 to 874
Iodine absorption .. ..	55 to 61	9
Saponification equivalent ..	286 to 292	209 to 228
Volume of $\frac{N}{10}$ alkali required by distillate from 5 grms. ..	0.5	7.0
SEPARATED FATTY ACIDS:—		
Plummet gravity at 99° C. ..	838 to 840	844
Iodine absorption .. ..	61 to 64	15.01
Mean combining weight ..	278	200

The most accurate determination of the cocoa-nut oil is obtainable from the saponification equivalent, as this estimation is practically unaffected by the presence of cotton-seed oil or tallow. Taking the average saponification equivalent of lard at 289, and that of cocoa-nut oil at 219, there is a difference of 70, and hence every 0.70 of fall in the equivalent below 289 indicates the probable presence of 1 per cent. of the adulterant. Comparatively small proportions of cocoa-nut oil in lard can be detected and safely certified.

In conclusion, I may add that some time since I received very pressing inquiries from America as to where cocoa-nut stearin could be obtained, but was compelled to reply that it was not now in the market. Some months ago I met with a butter which was undoubtedly adulterated with cocoa-nut oil.

101, Leadenhall Street, E.C.

September 19th, 1888.

#### THE REMOVAL OF IODATE FROM THE IODIDE OF POTASSIUM BY MEANS OF ZINC AMALGAM.

By H. N. MORSE AND W. M. BURTON.\*

THE frequent occurrence of the iodate of potassium in the commercial iodide, and the difficulty of removing it, are two of the most serious obstacles in the way of the free use of the iodometric method, either for quantitative work, or for the detection of oxidising agents. The fact that so many methods have been proposed for the preparation of iodide free from the iodate, and for the removal of the latter from the former, indicates that a simple and effective method for the preparation of the pure iodide of potassium is much needed, but not readily found.

In the course of our work upon the atomic weight of zinc it became necessary for us to prepare some iodide of potassium free from iodate, in order to test for the presence of the oxides of nitrogen, which Marignac† supposes to remain in the oxide of zinc prepared from the nitrate, even up to the dissociating temperature of the oxide. The method which we employed for the purpose consists in boiling the solution of the iodide with zinc amalgam. By this means the iodate is completely reduced with formation of zinc hydroxide, and the filtered solution is found to be free from both mercury and zinc. It is recommended to make the amalgam quite rich in zinc, and to have the filter paper through which the hot solution of iodide is to be filtered saturated with boiling water.

\* *American Chemical Journal*.

† Archives des Sciences Phys. et Nat (3) 10, 195.



The efficacy of the method was tested upon solutions of pure iodate of potassium. In one case one gram. of the iodate dissolved in 50 cubic centimetres of water was completely reduced within forty-five minutes; in another, two grams. dissolved in the same amount of water were reduced within one hour and a quarter. The bromate and chlorate of potassium are also reduced by zinc amalgam, but much more slowly than the iodate. Of the two, the chlorate reduces less rapidly than the bromate.

The zinc amalgam used in the reduction is best prepared by agitating zinc dust with mercury in the presence of tartaric acid, and washing with water.

#### A METHOD FOR THE ANALYSIS OF BUTTER, OLEOMARGARINE, ETC.

By H. N. MORSE AND W. M. BURTON.\*

THE method which we here describe has been in use in this laboratory during the past year. Its advantages are: 1st. That it is volumetric throughout; 2nd. That it obviates the necessity of weighing the specimen of fat; 3rd. That it readily discriminates between genuine butter and any mixture of cocoanut oil and other fats or oils. That is, it succeeds at that point where the methods of Hehner and of Koettstorfer may fail.

It depends upon the fact that the relative quantities of alkali required to neutralise the soluble and insoluble acids are for any one fat or oil quite constant, but for different fats or oils quite variable.

The following statement contains the relative percentages of alkali required to neutralise the soluble and insoluble acids of butter, cocoanut oil, cotton-seed oil, oleomargarine, lard, and beef tallow:—

	Per cent. KOH required for insoluble acids.	Per cent. KOH required for soluble acids.
1. Butter .. .. .	86.57	13.17
2. Cocoanut oil (unwashed) .. ..	91.95	8.17
3. Cocoanut oil (washed with hot water) ..	92.43	7.42
4. Cocoanut oil (washed with dilute $\text{Na}_2\text{CO}_3$ ) ..	92.33	7.45
5. Cotton-seed oil .. .. .	92.05	7.76
6. Oleomargarine .. .. .	95.40	4.57
7. Lard .. .. .	95.96	3.82
8. Beef tallow .. .. .	96.72	3.40

There is, between the amounts of alkali required to neutralise the soluble acids of butter and of unwashed cocoanut oil, a clear difference of 5 per cent.

Evidently it would not be possible to make any mixture of the other substances than butter in this list which would not show even a greater divergence from butter in respect to the soluble acids. The only way in which the proportion of soluble acids can be increased beyond 8.17 per cent. is by the addition of butter itself.

It was found by Moore that a mixture of butter 50 per cent., oleomargarine 27.5 per cent., and cocoanut oil 22.5 per cent., could not be distinguished from butter either by the method of Hehner or by that of Koettstorfer. Such a mixture gave us:—

Per cent. KOH required for insoluble acids.	Per cent. KOH required for soluble acids.
90.17	9.70

\* *American Chemical Journal.*

There is still a difference in respect to soluble acids of 3.47 per cent. between such a mixture and butter; and it would not be practicable to so far increase the proportion of butter that there would not still be a perceptible difference in this respect between genuine butter and the mixture. The figures thus far given are mean quantities; we give in the following table the individual results from which they were calculated. The first column contains the number of milligrams of potassium hydroxide required to saponify one gram. of fat; the second and third columns, the number of milligrams required to neutralise the insoluble and soluble acids contained in one gram. of the fat; while the fourth and fifth give the percentages corresponding to the quantities in two and three.

A. *Butter.*

	I. Mgs. KOH for 1 gram. fat.	II. Mgs. KOH for insoluble acids.	III. Mgs. KOH for soluble acids.	IV. Per cent. KOH for insoluble acids.	V. Per cent. KOH for soluble acids.
1.	230.39	199.57	30.21	86.62	13.11
2.	231.14	199.83	30.94	86.45	13.38
3.	230.66	200.11	30.07	86.75	13.04
4.	230.71	199.67	30.54	86.53	13.23
5.	230.94	199.85	30.29	86.53	13.11
Mean, 86.57					13.17

B. *Cocoanut Oil (unwashed).*

1.	266.88	245.50	21.91	91.98	8.20
2.	266.96	244.87	22.01	91.72	8.24
3.	266.58	245.68	21.53	92.15	8.07
Mean, 91.95					8.17

*Cocoanut Oil (washed with hot water).*

4.	262.44	243.17	19.11	92.65	7.28
5.	263.33	242.84	19.91	92.21	7.56
Mean, 92.43					7.42

*Cocoanut Oil (washed with dilute Na<sub>2</sub>CO<sub>3</sub>).*

1.	263.73	243.27	19.91	92.24	7.54
2.	263.42	243.39	19.31	92.39	7.33
3.	263.97	243.81	19.57	92.36	7.41
4.	264.22	243.96	19.50	92.33	7.38
5.	263.82	243.61	20.03	92.33	7.59
Mean, 92.33					7.45

C. *Cotton-seed Oil.*

1.	200.23	184.31	15.57	92.04	7.77
2.	200.54	184.04	15.71	91.77	7.83
3.	199.74	184.26	15.06	92.25	7.54
4.	199.81	183.72	15.36	91.95	7.68
5.	199.65	184.18	15.92	92.25	7.97
Mean, 92.05					7.76



D. *Oleomargarine.*

202.70	193.02	9.31	95.22	4.59
202.61	193.46	9.01	95.48	4.44
202.40	193.21	9.71	95.45	4.79
202.62	193.65	9.21	95.57	4.54
202.83	193.27	9.12	95.29	4.49
Mean, 95.40				4.57

E. *Lard.*

199.77	191.78	7.31	96.00	3.65
198.93	191.11	7.91	96.06	3.98
199.54	191.29	7.71	95.86	3.94
199.75	191.62	7.87	95.93	3.94
199.40	191.34	7.17	95.97	3.59
Mean, 95.96				3.82

F. *Beef Tallow.*

200.44	193.43	6.76	96.50	3.37
199.70	193.20	6.92	96.74	3.46
200.17	193.81	6.47	96.82	3.23
199.60	193.13	6.91	96.76	3.46
199.84	193.42	7.01	96.78	3.50
Mean, 96.72				3.40

G. *Mixture: Oleomargarine 27.5; Coconut Oil 22.5; Butter 50 per cent.*

231.	208.77	22.65	90.37	9.81
230.82	208.13	22.21	90.16	9.62
231.15	208.11	22.76	90.04	9.84
231.36	208.51	22.16	90.12	9.57
Mean, 90.17				9.70
Percentages for pure butter, 86.57				13.17
Difference, 3.60				3.47

(To be continued.)

## SOURCES OF ERROR IN DETERMINATIONS OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. ATWATER.\*

object of this paper, which concludes a series upon the same general subject, describe some further experiments in this laboratory, adduce results of observations where, and mention certain conclusions that seem warranted concerning sources of error in the determination of nitrogen in protein compounds, especially those of animal origin, by soda-lime, and the means by which they may be obviated.

Leaving out of account the apparent gain of nitrogen which may come from impure soda-lime, or from obvious errors in manipulation, to be mentioned beyond, the principal sources of error involve loss of nitrogen. Among the sources of loss are:

\* *American Chemical Journal.*

1. Incomplete ammonification of the nitrogen.
2. Dissociation or oxidation of the ammonia formed.
3. Failure of the ammonia to be completely caught and retained by the acid solution.

*Loss by Incomplete Ammonification of Nitrogen.*

Four ways suggest themselves by which nitrogen may fail to be changed into the form of ammonia and may hence be lost in soda-lime determinations, namely, by :

1. Incomplete decomposition of the nitrogenous substance, part of the nitrogen remaining in the partially decomposed (charred) residue.
2. Formation of compounds, *e.g.*, cyanides, which remain in the tube.
3. Escape of nitrogen in the free state.
4. Formation of volatile nitrogenous products other than ammonia, which either pass through the acid solution unabsorbed, or if absorbed, are not detected by the subsequent titration with alkali or other means used to find the amount of nitrogen in the solution.

As no especial study of the first three of these sources of loss has been made here, I need only refer to them briefly.

To insure complete decomposition, fine pulverisation, thorough mixing with soda-lime, and heating, at sufficiently high temperature, until no charred residue remains, would seem to be the requisites. As to the fineness of pulverisation needed there seem to be differences of opinion among chemists. Ritthausen\* and Gruber†, for instance, lay great stress upon it. Gruber cites comparative determinations of nitrogen in flesh in which the incorrect results fell nearly 0.9 per cent. below the correct ones, and the only difference in the conduct of the analyses was that, in the defective ones, the "Fleischpulver noch einzelne wahrnehmbare Körnchen enthielt." Johnson and Jenkins, on the other hand, say that : "Contrary to what is commonly stated, fine pulverisation of the substance to be analysed is not necessary. If the substance will pass holes of one millimetre in diameter it is fine enough. A sample of dried blood which passed through a sieve with meshes one millimetre in diameter gave 7.58 per cent. of nitrogen. A portion of the same, ground extremely fine with sand, gave 7.64 per cent. Fish-scrap passed through the same sieve gave 8.98 per cent. of nitrogen ; when ground with sand, 8.95 per cent. A second sample of fish, sifted as above, gave 8.69 per cent. of nitrogen. By the absolute method it yielded 8.79 per cent."‡

The not inconsiderable experience of this laboratory confirms very decidedly the opinion last quoted. We have for a number of years used a sieve with circular apertures of one millimetre diameter. A large number of comparisons of results obtained by the soda-lime and absolute methods, and in a considerable number of cases with that of Kjeldahl, have given results so concordant as to persuade us that particles which have passed through this sieve are fine enough.

In absence of definite statements as to the actual fineness which is regarded as desirable by those who insist upon very fine pulverisation, I am inclined to suspect that the standard adopted by Johnson and Jenkins, and in this laboratory, would perhaps be

\* Jour. prakt. Chem. 116, 1874, p. 17.

† Ztschr. f. Biol. 16, 380.

‡ Report of Conn. Agl. Expt. Station, 1878 116



regarded as satisfactory in respect to fineness by them and by investigators generally. Certain it is that when, as in a number of instances, we have tried to use a sieve of one half millimetre aperture, we have found the process of grinding painfully laborious and time-consuming, even with the use of a mill especially devised for the purpose and exceeding in convenience all other forms which I have seen.

As to formation of cyanogen or other compounds which would be retained by combining with the bases of the soda-lime, I have been unable to find any experimental facts which would lead to the supposition that there is any considerable danger of loss in this way in the combustion of ordinary animal and vegetable substances, provided the soda-lime contains enough water, and the quantities of soda-lime and substance, temperature, and other conditions are appropriate.

Regarding the escape of free nitrogen by decomposition of such nitrogenous compounds as those in question (leaving out of account the dissociation or oxidation of ammonia formed from them), I am likewise unable to find any facts which imply special danger of loss in this way, although it might, perhaps, be feared from such observations as that of Liebermann that albumin on being heated with caustic baryta to  $150^{\circ}$  yields free nitrogen.\* The main assurance that it does not escape to any great extent when the operation is rightly conducted, is found in the agreement of the results obtained with those obtained by other methods.

The presence of nitrates, as impurities in soda-lime or otherwise, may cause loss of nitrogen, perhaps as free nitrogen, but this is of course simply a contingency to be feared from careless work.

*Loss of Nitrogen by Formation of Volatile Products other than Ammonia.*

As long ago as 1860 Mulder called attention to the danger of loss by formation of volatile products which, escaping ammonification within the tube, were either not caught by the acid or, if retained, escaped determination by either titration with alkali or precipitation by platinic chloride.† About the same time Strecker observed that guanidine salts failed to yield all their nitrogen as ammonia with soda-lime.‡

Ritthausen and Kreussler were unable to get more than 7.9 per cent. of nitrogen from leucine by soda-lime alone, even when the greatest care was taken to pulverise and mix it with the soda-lime, and 120 times as much soda-lime as substance was used. Adding sugar, however, they got as high as 10.43 per cent., very nearly the theoretical amount, and Ritthausen suggests that the trouble with leucine, as with other compounds, may be that volatile cleavage products are formed and pass over the soda-lime without their nitrogen being changed to ammonia.§

Mäckerer insists that aniline-like products may be given off instead of ammonia, and finds proof of this in observations that some substances, as for instance gluten and leucine, gave nearly or quite their full amount of nitrogen with platinic chloride, while there was decided loss by titration with alkali. With blood albumen and horse-flesh, however, there was a loss by titration which the platinic chloride did not amend, and

\* Jlt. Ag. Chem. 21, 766. See also Wanklyn and Gamgee, J. Chem. Soc. 21, 1868, 25.

† Chem. Centrbl. 1861, 44.

‡ Ann. Chem. (Liebig) 118, 1861, 161.

§ J. prakt. Chem. 111, 1871, 310.

which can be explained by assuming that products were formed which are not precipitated by the platinic chloride, which does precipitate aniline and kindred compounds.\*

Among the materials in which Nowack and Seegen failed to obtain the normal quantity of nitrogen (see beyond) by soda-lime was barium kynurenate. Gruber very aptly remarks that as kynurenic acid has been shown to be a derivative of quinoline, the failure may have been due to distillation of the latter.†

E. Salkowski suggests that in determinations of albuminoids by soda-lime, bases of the pyridine series may be formed and escape ammonification. He also suggests that a substance with red colour which he obtained in burning at low heat with a small quantity of soda-lime may perhaps be the chromogen of urobilin.‡

Kjeldahl in the discussion of his method for nitrogen determinations, referring to Mulder's advice to avoid any large amount of vacant space inside the combustion tube, states a very interesting observation of his own. In making combustions of quinine-hydro-chloride with soda-lime, he found that when the soda-lime was loosely packed and shaken so as to leave a channel in the ordinary way, he obtained only about one half the nitrogen, while with the conditions in every respect the same except that the tube was well packed with soda-lime so as to leave a minimum of vacant space, he obtained practically the whole of the nitrogen as ammonia by titration. He attributes the loss in the first instance to the formation of volatile nitrogenous products which, not being brought into intimate contact with the soda-lime, escaped ammonification §

(To be continued.)

## ON THE RELATIVE VALUE OF DIFFERENT PEPSIN TESTS.

BY JAMES H. STEBBINS, JR.¶

THE gastric juice, secreted by the peptic glands of the stomach, contains two important principles—viz., hydrochloric acid and a ferment called pepsin.

By the combined action of these two principles it performs its physiological function, which consists in digesting albuminoid matters; i.e., it disintegrates, dissolves, and converts them into soluble, non-coagulable products, known as peptones.

Wasmann first isolated pepsin in 1839, but it must be said that his process, as well as those which have since been proposed for the preparation of this article, do not furnish perfectly pure pepsin. The product obtained is always more or less mixed with extractive matter, mineral matter, and peptone, sometimes in considerable quantity, which our present knowledge of the art has not enabled us to completely remove. Perfectly pure pepsin is, therefore, not known, but we are nevertheless enabled to prepare products of very high digestive power.

Many methods have been proposed for testing pepsin, but none of them are perfectly satisfactory.

Thus, Bidder and Schmidt place a known weight of small cubes of coagulated white of egg in contact with a liquid containing a known weight of pepsin, dissolved in

\* Archiv Physiol. (Pflüger) 8, 1874, 207.

† Ztschr. f. Biol. 16, 1880, 377.

‡ Ztschr. anal. Chem. 16, 261 and 408.

§ Ztschr. anal. Chem. 22, 1883, 380. See also Arnold, Ber. d. chem. Ges. 18, 1885, 809.

¶ Journal of the American Chemical Society.



hydrochloric acid of 0.2 per cent. strength, and heat the mixture for about five hours at 50° C. At the end of this time the non-dissolved albumen is washed and weighed. The loss in weight of this albumen indicates the digestive power of the pepsin.

This method of assay has the inconvenience of taking into account only the amount of albumen dissolved, without paying attention to the amount of albumen really converted into peptone. The same applies also to the two following methods :—

Grünhagen allows fibrin to swell in hydrochloric acid of 0.2 per cent. strength, and places the thick jelly thus obtained in a funnel, closed at one end with a little glass wool, and exposes the whole in a drying oven to a heat of 45° C. After all excess of water has drained off, a certain number of drops of a solution, containing the pepsin under examination, is allowed to trickle upon the jelly contained in the funnel. In about two minutes the liquid will begin to drop from the funnel, and the rate of dropping in a given time is said to be proportional to the strength or activity of the pepsin.

P. Grützner determines the value of pepsins by allowing flocks of fibrin, stained with carmine, to digest under similar conditions, and then estimating colourimetrically. At the end of a certain time, the intensity of the colouration of the liquid separated by decantation from the non-dissolved fibrin, by comparing it with a certain number of standard solutions of carmine. It is very easy to stain fibrin evenly with carmine, and therefore the intensity of the colouration of the solution is proportional to the amount of fibrin dissolved by the pepsin.

The methods I propose to discuss in this paper are three; viz, the U. S. P. test, the Manwaring test, and the Kremel test.

According to the experiments of numerous investigators, the peptic digestion of albuminoids depends upon several conditions.

1. The temperature.

The pepsin of fish acts energetically at 20° C., but the pepsin of mammals requires a higher temperature, and it has been found that peptonisation is most active between 40° C.—50° C. Above this, digestion runs much slower, and ceases totally towards 60°—80° C.

2. The quantity of pepsin.

There being no such thing as absolutely pure pepsin, it has been impossible to determine with accuracy the amount of albumen which can be converted into peptone by a given quantity of the ferment. We know only that the amount is very large, provided that from time to time a little acid and water is added in order to maintain a certain degree of dilution.

The quantity of albuminoid which can be digested in a given time increases rapidly with the quantity of pepsin employed till it reaches a maximum, and then decreases slowly. The quantity of peptone finally obtained increases with the proportion of pepsin.

3. The quantity of water.

As the products of digestion accumulate the rate of peptonisation gradually decreases. The addition of a fresh quantity of acidulated water causes the peptic action to recommence, until it has reached a certain limit, beyond which the reaction ceases entirely.

4. The nature and quantity of the acid used.

A large number of acids may take the place of hydrochloric acid in peptic digestions, but none of them are as efficient as the latter. A. Mayer found that with the use of hydrochloric acid complete peptonisation occurred in from 3 to 5 hours, with nitric acid in about 5 hours, with oxalic acid in 13 hours, and with sulphuric acid in 19 hours.

According to Brücke, peptonisation is already very active in a medium containing only 0.8 pts. of hydrochloric acid per 1,000, and attains its maximum with a concentration of 1 pt. of acid in 1,000 of water. A too large proportion of acid hinders peptonisation, 7 pts. of acid per 1,000 of water being sufficient to make the action very slow. Mayer thinks that the most favourable proportion of acid is 2 pts. per 1,000 water, or 0.2 per cent.

5. The time of action.

6. The variety and character of the albumen.

One of the most largely used tests in this country is the U. S. P. test, which reads as follows:—

"One pt. of saccharated pepsin, dissolved in 500 pts. of water, acidulated with 7.5 pts. of hydrochloric acid, should digest at least 50 pts. of hard-boiled egg albumen, in 5 or 6 hours, at 100—104° F. (37.5—40° C.)"

The above test seems simple, but in reality it is unreliable and misleading, as no two persons using the same pepsin can obtain the same or even approximate results; it is, therefore, not surprising that we meet with such a diversity of conclusions.

The weak points in the above test are the following:—

1. The test is based upon the amount of albumen which can be dissolved in a given time (including peptone and intermediary products), but does not take into consideration the amount of peptone actually formed, and this I claim to be of the greatest importance.

2. It directs that a given pepsin shall digest at least 50 pts. of coagulated albumen. Now, in order to determine how much albumen has actually been dissolved, it is necessary to use an excess of albumen, and then weigh what remains undissolved. The test in question does not specify how much albumen shall be used, but leaves it entirely to the option of the experimenter. I consider this to be a weak point, as it makes quite a difference whether only a small or large quantity of albumen is used.

3. It is difficult to see how accurate results are to be obtained by weighing the amount of undissolved albumen remaining after a digestion, because it is impossible to find two samples of coagulated albumen which contain exactly the same quantity of moisture; and besides this, the quantity of moisture is very liable to vary during the weighing, owing to the loss of moisture by evaporation.

4. It is not stated how long the eggs shall be boiled! This is a very important matter, as digestion differs greatly according to whether the eggs are boiled for a short or a longer time.

5. No provision is made for the size of the pieces of coagulated albumen. This, also, is very important, as it has been found that the greater the surface of the albumen exposed to the peptic ferment, the greater will be the amount of albumen digested.

6. This test applies only to saccharated pepsins, and no provision is made for other brands of pepsin.

It will, therefore, be seen that the U. S. P. pepsin test is absolutely unreliable and misleading.

*(To be continued.)*



## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

**FLOUR IN SAUSAGES.** H. TRILLICH. *Zeitschr. f. angew. Chemie*, No. 17.—The idea that it is possible to introduce a large amount of water in sausages by means of flour is so shared by Dammer, in his *Lexicon der Verfälschungen*. Schmidt and Mühlheim, in their work on meat and meat preparations, say 1 part of flour is capable of holding 27 parts of water. So a sausage may look all right, but still contain, say, 67 per cent. of water. The author proved, last year, that a small quantity of added flour has no influence on the quantity of water the sausage is capable of holding. If the water is calculated from the amount of added flour the result is, of course, too low, and the true amount is got by adding the water combined with the meat. If the flour is capable of holding 67 per cent. of water, then, as the average percentage of water in fresh meat is 64, the total percentage of water will be—

$$67 + \frac{27 \times 64}{100} = 84.3 \text{ per cent.}$$

The author's analyses of Münchener sausages sometimes showed 76.5 per cent. of moisture; but even if they also contained 6 per cent. of flour their appearance soon betrayed them. A sausage with 83.84 per cent. of water is, in fact, not a saleable article.

If, however, the 27 per cent. of meat means dry substance, then it will be impossible to pretend that all the water is in combination with the flour; then 27 per cent. dry meat represents 75 per cent. fresh meat with 64 per cent. of moisture, and therefore 48 per cent. may be taken as the natural amount of water in the used meat. The remaining 19 per cent. must have been introduced in the sausage by some device or other; but it is not necessary to use flour for this purpose. The water absorption (binding) power of meat is 25.3 per cent. Supposing it to contain less natural moisture—say only 50 per cent.—it would not, as may be supposed, have a larger absorption power, but, as experience has taught, rather a smaller one.

*Calculation of the water absorption power of sausage meat.*—Taking the moisture of meat to be 60 per cent., then, if  $a$  represents the moisture of the sausage, and  $s$  the amount of added flour, the absorption power will be—

$$y = a - 1.5 (100 - a - s) \text{ per cent.}$$

For a different percentage of moisture in the meat, replace 1.5 by  $\frac{a}{100 - a}$ .

The absorption power for the starch-free article will be—

$$z = \frac{( \mid 100 - s \mid - 2.5 \mid 100 - a - s \mid )}{2.5 \mid 100 - a - s \mid}$$

$$2.5, \text{ if required, to be replaced by } \mid 1 + \frac{a}{100 - a} \mid$$

To get trustworthy results, examine the sausage when quite fresh. L. DE K.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his correspondents.]

## POLLUTED DRINKING WATER AND THE CLOSURE OF WELLS.

To the Editor of the ANALYST.

SIR, — In my paper on this subject, read at the meeting of the Society of Public Analysts on June 20th, 1888, reference is made to the case of a well at Enfield, the water of which was pronounced by one chemist as "containing sewage, and totally unfit for drinking purposes," while another, whose analytical results were essentially the same, reported it "a perfectly safe water to use for drinking and domestic purposes, being free from all traces of sewage contamination."

Owing to this conflict of evidence the magistrate referred the case to Dr. Bell, of Somerset House. In my paper I say, "singularly enough, two other chemists, whose analytical results agree with Mr Lloyd's, gave favourable reports of the water."

Dr. Bell, in a letter to me, complains that this remark entirely misrepresents the terms of his report. While disclaiming, as I hope is hardly necessary, any intention to misrepresent Dr. Bell's views, I may state that I made the remark without having seen the report, and on the strength of a paper read by Mr. Lloyd at a meeting of the Society of Medical Officers of Health, and the report of this in the *British Medical Journal* of May 26th. The dismissal of the case by the magistrates after receiving Dr. Bell's report, confirmed in my mind the impression made upon it.

Dr. Bell has sent me the following extract from his report:—

"The organic impurity present, as indicated both by the albuminoid ammonia and the oxygen consumed, does not exceed by an appreciable extent the limit allowable in a potable water.

"From the large quantity of saline matters present in the water, including nitrates, chlorides, and salts of ammonia, it is evident that it passes through a stratum of earth largely charged with sewage or organic refuse substances, and carries down with it various products resulting from the oxidation of the organic matter.

"These products, although not absolutely dangerous to health, are very objectionable in water intended for domestic purposes, and when present to the extent in which they exist in the water under consideration, we should not recommend it for use for potable purposes.

"Although, therefore, we are unable to entirely agree with the opinion pronounced by either analyst, we should advise the discontinuance of the use of the water on account of its unsatisfactory and doubtful character."

It is clear from the last two paragraphs of the report that, though Dr. Bell does not condemn the water as dangerous, he does not give that favourable report of it which the information at my command led me to understand.—I remain, Yours faithfully,

ALFRED HILL.

Borough of Birmingham Health Department, The Council House,

September 22nd, 1888.

TEST FOR THE PURITY OF MILK.—The fashion of empirical tests has quite set in and the following paragraph has been going the round of the presses:—"A very simple and ingenious method of testing whether milk has been adulterated with water has been announced by a German scientist, who says it will detect the presence of the smallest quantity of the objectionable 'Simpson.' The plan recommended is to place the milk in a deep vessel, and procuring a highly polished knitting needle, to insert it vertically in the doubtful fluid. The needle must then be steadily withdrawn, and, if a drop remains at the end, then the milk is pure. If, on the other hand, the drop is not found, it may be safely concluded the milk has been adulterated. This is certainly a very simple, and it is said to be a reliable test." If we are not mistaken we have heard of this test twenty years ago.



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### SPECIFIC GRAVITY AT 100° F. OF FAT WHEN CLEAR, AND ALSO WHEN CLOUDED WITH CRYSTALS.

BY E. W. T. JONES, F.I.C.

SOME time ago I was taking the specific gravity of some lards at 100° F. with a bottle, when some of the samples, through being left just about 100° F. for some hours, became more or less granulated or clouded with crystals; they were still fluid enough to pour into the bottle, and upon taking the specific gravity in this state I found them all unduly high, and I became suspicious, the abnormally high specific gravity was due to the crystalline state, hence I made the following experiments, which prove it to be so.

A sample of genuine lard was melted quite clear, and then the temperature allowed to fall whilst stirring with a thermometer till the proper temperatures was attained, and the specific gravity at 100° F. taken. The fat remaining quite clear, it gave

(i.) 905.87

(ii.) 905.72.

I now allowed this melted fat to remain at the temperature, about 100° F., for some hours, by which time it had become somewhat granulated or clouded with crystals, then carefully adjusting the temperature, I took its specific gravity at 100° F., still in its clouded condition, getting

(i.) 910.49

(ii.) 910.30,

this shows the increase due to its crystalline state. I then took another sample of lard, this was softer, and did not granulate to the same extent as the former one, and so the difference in the specific gravity of the clear and clouded fat is not so great.

Clear, 906.10

Clouded, 908.47.

I do not suppose recording these facts will be of much practical use to Public Analysts, but from a scientific point of view they are very interesting.

## A METHOD FOR THE ANALYSIS OF BUTTER, OLEOMARGARINE, ETC.

BY H. N. MORSE AND W. M. BURTON.

*(Concluded from page 193.)**The Reagents.*

1. A solution of hydrochloric acid of such strength that one cubic centimetre of it is equivalent to 20 milligrams. of potassium hydroxide.
2. A solution of hydrochloric acid one-tenth as strong as the first.
3. A solution of potassium hydroxide in 95 per cent. alcohol approximately equivalent to the first acid.
4. A solution of potassium hydroxide in 95 per cent. alcohol one-tenth as strong as the preceding.

*The Mode of Procedure.*

The dry and filtered fat is well stirred during solidification, to prevent the separation of the lower from the higher melting constituents.

Any convenient quantity of the material, between one and two grams., is placed in an Erlenmeyer flask having a capacity of 250 cubic centimetres, and treated with that amount of alkali which is found to be equivalent to 40 cubic centimetres of the acid No. 1. The flask is then placed upon the water-bath, and heated to the boiling point of alcohol for twenty minutes.

The excess of alkali is determined by acid No. 1, with the use of phenolphthalein as the indicator. Thus the number of milligrams. of KOH required for the neutralization of all of the acids in the fat is found. The flask is then returned to the water-bath, and heated until all of the alcohol has been expelled. During the evaporation of the alcohol, the soap, which in the first place was neutral, becomes alkaline, owing doubtless to the evaporation of a small amount of volatile acids in the form of ethers; but the loss from this cause is very slight.\* When the odour of alcohol is no longer perceptible the soap is treated with that quantity of acid No. 1 which is necessary to exactly liberate all of the acids contained in it. This quantity is, of course, the difference between 40 and the number of cubic centimetres required to neutralise the excess of the alkali used in saponification. The flask is again returned to the water-bath, and heated until the liquid becomes clear. But, in order to prevent loss of volatile acids, it is now supplied with a stopper carrying a glass tube about 5 millimetres in diameter, and 400 millimetres in length. The upper end of the tube is bent downwards, and to it is attached a small U-tube containing a few cubic centimetres of water. The contents of the flask are filtered through double thick paper which has been wet with hot water, and then washed with boiling water until the volume of the filtrate equals a litre; care being taken to wash thoroughly the flask itself. The contents of the condensing arrangement are washed into the filtrate. The funnel containing the washed insoluble acids is placed in the flask in which the saponification, etc., were effected, the paper pierced, and the contents washed through with hot 50 per cent. alcohol.

\* This loss may be made good by bringing the solution to a neutral condition by means of very dilute acetic acid. The quantity of acetic acid thus introduced is, of course, equivalent to the acid which has been lost during the evaporation of the alcohol. We are under the impression that there is no such loss of volatile acids during saponification, owing to the presence of a considerable excess of *alkali*.



We thus have the soluble and insoluble acids separated, and it only remains to determine the amount of alkali required to neutralise each. The soluble acids can be directly determined by the weaker solution of the alkali; but to determine the insoluble acids it is better to add the quantity of alkali which was required for the saponification, and, after warming upon the water-bath, to titrate back with the weaker acid. It will be observed that a determination of the insoluble acids alone by this method suffices to distinguish butter from other fats and mixtures, and that if these only are to be determined it is not necessary to use the condensing arrangement. Moreover, it is not necessary to use the condensing arrangement. Moreover, it is not necessary to know the exact strength of the solutions employed, since a knowledge of the relation of the acids to each other and to the alkalies is all that is needed to enable us to determine, in the fat, the relation of the soluble to the insoluble acids. Hence it is practicable to dispense with the use of the balance altogether. It is frequently recommended to make blank determinations, in order to control the error introduced by the action of the alkali upon the glass of the flasks. But a little reflection will convince one that this practice may itself be the cause of considerable error, inasmuch as the extent to which the glass is affected by the alkali depends upon its quality and upon the treatment to which it has previously been subjected. In order that the results of the blank may be applied to a correction of those of the determinations, it is necessary that the glass employed in the two cases should be the same in respect to composition and previous use in the laboratory. It is better, according to our experience, to discard the use of blanks, and to boil out with strong alkali all flasks employed in such work. The subsequent action of dilute alkali upon glass which has been treated this way is very slight. The water and alcohol employed in making the standard solutions should, of course, be thoroughly dried, and afterwards protected from the carbon dioxide of the oil by keeping them in bottles or flasks, which are supplied with stoppers carrying tubes filled with soda-lime. The use of blanks is then not necessary on account of any carbonic acid contained in the solution.

#### LARD: ITS ADULTERATION WITH COTTON-SEED OIL AND DETECTION THEREOF.

By MICHAEL CONROY.\*

The purity of lard for use as an ointment basis is a matter of some importance, and consequently the British Pharmacopœia directs it to be made from the perfectly fresh internal fat of the abdomen of the hog; and, according to the same authority, it should respond to the following characters and tests: "A soft, white, fatty substance, melting at 100° F. (37·8 C.); has no rancid odour; dissolves entirely in ether. Distilled water, which it has been boiled, when cooled and filtered, gives no precipitate with nitrate of silver, and is not rendered blue by the addition of solution of iodine." These tests are very good so far as they go, but they are, unfortunately, not sufficient for the misapplied genuinity of the present day.

It has recently been shown by the United States press that cotton-seed oil is used to an enormous extent for the adulteration of lard, which may be seen by the opposition

\* *Pharmaceutical Journal*.

given to a Bill introduced into Congress for the prevention of this adulteration. The Mississippi Legislature passed resolutions against the Bill; the merchants and Cotton Exchanges of New Orleans sent protests to Congress against it; while the *New Orleans Times* calls it a Bill to reduce the value of cotton-seed 50 per cent. The following tit-bit is from the *Nation*: "We cannot see any difference between this Bill and one which should seek to put a clog on new inventions. The discovery that butter can be made from the fat of a slaughtered ox as easily as from the cream of a living cow was a great boon to mankind, and one which cannot be suppressed, although it may be temporarily crippled by legislation. The discovery that the sun's light and heat work the same result in the production of edible fats in the seed of the cotton plant as in the fruit of the olive or in the bodies of swine is akin to it, and is likewise a benefit to the human race. Why should Congress undertake to prevent the diffusion of this blessing? If the proposed Bill does not prevent it, the measure will be useless to those who clamour for it."

The value of these quotations is apparent when we consider the vast quantity of American lard that is imported into this country. I have no statistics at hand, but I think it is under the limit to place it at one-half the total consumption, and we are told by the *American Oil and Drug Reporter* of April 4th last that "the pure food laws of England have in no way interfered with the sale of American refined lard, and no complaints have come to the surface, which is good evidence that the article gives satisfaction under the rigid scrutiny of exacting foreign buyers." Since this date, however, a change has come over the spirit of the *Reporter's* dream, for the matter has been vigorously taken up and several prosecutions have been carried, but as the adulteration is a paying one, it is not likely to be easily crushed.

Fortunately, the presence of cotton-seed oil in lard is easily detected, and my object in bringing the matter under your notice is to lay before you some of the tests that I have myself tried. The first is the nitric acid test, which some years ago I had the honour of reading a paper on before the Liverpool Chemists' Association, in connection with the adulteration of olive oil. It consists in heating and stirring well about half an ounce of lard with one-tenth its weight of strong nitric acid, specific gravity 1.42, in a porcelain dish of about 8 ounces capacity until brisk action commences, when it should be removed from the source of heat. Pure lard sets in about an hour to a pale orange-coloured solid, like citrine ointment, but if adulterated with cotton-seed oil it takes a more or less deep orange-brown tint, according to the extent to which that oil is present. There are two drawbacks to this test, the first being that lard sometimes contains a small amount of water, which reduces the strength of the acid, causing the acid to be less energetic, and thus leading to error; the second is that the difference in colour between pure and adulterated samples is not sufficiently definite when the adulterant is under 5 per cent.

I have also tried the test proposed by M. Labiche, who says that when cotton-seed oil is treated with subacetate of lead and caustic alkali, it gives almost immediately an orange-red reaction. The author mixes equal parts of the oil and a saturated solution of neutral acetate of lead and adds ammonia, stirring briskly. The acetate of lead decomposes and the nascent oxide reacts upon the oil, causing it to turn red. If 20 per cent. of cotton-seed oil be present the sample is said to turn red at once, lesser



quantities show after some time. In my hands this test has proved an utter failure, but I think it may be due to the fact that the cotton-seed oil which I used in my experiments was highly refined, and it is quite possible that the crude oil would give this reaction.

The next test is by Ernest Milliau, and was proposed by him for the detection of cotton-seed oil in olive oil. It is an excellent test and quite applicable to lard. It is as follows:—In a porcelain capsule, holding about 1 litre, 15 c.c. of the sample in question are heated to  $110^{\circ}$ . Then, whilst still continuing the heat, we pour upon the oil a mixture of 15 c.c. of a solution of soda of  $40^{\circ}$  Baumé in distilled water and of 15 c.c. of alcohol of 92 per cent. When the mass has become homogeneous we add, drop by drop, so as not to cool the paste and form clots, about half a litre of distilled water. After boiling for a few minutes the fatty acids are separated by means of pure sulphuric acid diluted to one-tenth. As soon as the separation is complete and the sulphuric acid is in slight excess, 5 c.c. of the hydrated fatty acids are collected with a silver spoon and poured at once into a test tube, about 3 c.m. in diameter and 12 in length. We add 20 c.c. of alcohol of 92 per cent., and heat slightly in the water bath to dissolve the fatty acids. When the solution is effected, 2 c.c. of a solution of silver nitrate (3 grams in 100 c.c. of distilled water) are added, the tube is placed in a warm bath and heated until about one-third of the mass is evaporated. The tube is then removed from the water bath. Whatever the origin of the sample, its fatty acids remain unaltered if the sample be pure. But if cotton-seed oil is present the silver is reduced and blackens the fatty acids, which rise to the surface. In this manner 1 per cent of cotton-seed oil can be detected in olive oil.

In using this test for lard instead of for olive oil, as intended by its author, a brown colouration instead of a black one is obtained in samples containing cotton-seed oil, while pure samples remain perfectly white, and I find it better, instead of adding the half-litre of cold water, "drop by drop," to use boiling water. For delicacy and reliability this test leaves nothing to be desired, and its only drawback is the time it takes to perform.

Another test for the detection of cotton-seed oil in olive oil, dependent upon the nitrate of silver reaction, is given by Bechi as follows:—5 c.c. of the oil are mixed with 25 c.c. of 98 per cent. alcohol, and 5 c.c. of silver nitrate solution (prepared by dissolving 1 gram. of the nitrate in 100 c.c. of 98 per cent. alcohol), the mixture is heated to  $84^{\circ}$  C. If cotton-seed oil be present, the mixture becomes coloured, but not so if the oil be genuine. It is necessary to avoid heating by the direct flame, as other oils which may be present, such as linseed, colza, etc., will give colourations.

This, unlike the previous test, is not quite suitable for the detection of cotton-seed oil in lard, because lard sometimes contains traces of sodium carbonate, due to the fact that this substance is commonly used in washing lard that has become rancid. Slight traces of sodium carbonate decompose the silver nitrate, and the subsequent heating reduces it, causing samples of genuine lard to become darkened in such a manner that they might possibly be condemned as impure.

For several weeks past I have tried these and other tests with the object of finding

the most reliable and expeditious, and my experience is, that those dependent upon the reduction of silver nitrate are the best, and the following *modus operandi* has given me results that are entirely satisfactory and reliable, and only requires a few minutes time :—

1. Make a test solution containing 5 parts of silver nitrate and 1 part of nitric acid (specific gravity 1.42) in 100 parts of rectified spirit (specific gravity .838).

2. Melt a small quantity of the lard to be tested in a water bath and pour about 100 grains of it into a dry test tube, about half an inch in diameter. To this add 20 grain measures of the above-mentioned test solution, and place the tube in *boiling* water for five minutes, taking care that no water enters it.

Pure lard *remains perfectly white*, but if adulterated with cotton-seed oil, it assumes a more or less olive-brown colour, according to the amount present. The colour is best seen when the lard sets, and it saves time to put the test tube direct from the boiling water into a vessel of cold water.

The presence of 5 per cent. of cotton-seed oil in lard gives a very decided olive-brown colouration with this test, and 1 per cent. gives a colour quite distinct from genuine lard. The addition of nitric acid to the test solution is intended to neutralize any traces of alkali or alkaline carbonate that may be present, and it also prevents a slight reduction of the silver nitrate which takes place in genuine lard. It must not be forgotten that some samples of lard might possibly contain sodium chloride, though I have never met with any, in which case the silver nitrate would be precipitated as chloride instead of reacting on the oil, but this would be at once seen by the white curdy precipitate that would be formed.

## SYSTEMATIC EXAMINATION OF SULPHATE AND HYDROCHLORATE OF QUININE.

By C. HIELBIG.\*

THE examination of these quinine salts has caused quite a number of processes to be devised, but not one of these can be relied upon in furnishing a positive answer regarding the purity of these salts. To frame a method which allowed the presence or absence of the more frequently occurring impurities, such as quinidine, cinchonine and cinchonidine, to be proven in a simple and comparatively rapid manner, the majority of the published processes were carried out and their merits and defects ascertained; as the result the following compilation has been found to work successfully.

*For Sulphate.*

A. 1 gm. with 15 c.c.

*For Hydrochlorate.*

1 gm. and a solution of 0.4 gm. sodium sulphate in 1 c.c. water, with 30 c.c.

distilled water are agitated for five minutes, and filtered. To the filtrate is added 0.5 gm. Rochelle salt, agitated for five minutes, allowed to stand five minutes and filtered; the precipitate of tartrates is collected on a small filter and reserved, the filtrate for the

B. Detection of quinidine and cinchonine is divided into two portions, one of

\* *Pharm. Ztschr. f. Russl.*, 1888.



which is reserved; to the other add one drop of water of ammonia, and allow to stand for a few moments.

1. The solution remains clear; absence of quinidine and cinchonine, proceed E.

2. The solution becomes turbid; presence of quinidine and cinchonine or both; proceed C.

C. Detection of Quinidine. To the reserved portion (see B), add 0.5 gm. KI, shake for five minutes, allow to stand for same time. Observe either

1. The solution remains clear, if quinidine is absent; proceed D.

2. The solution becomes turbid or deposits tenacious resinous precipitate. In this case cinchonine must first be tested for according to D and then

a. In absence of cinchonine, the turbidity with KI indicates the presence of quinidine; proceed E.

b. In presence of cinchonine, the ammoniacal solution in B is filtered, the precipitate washed with distilled water and the thalleioquin reaction\* carried out with the precipitate. If the intense green colour is produced there is quinidine present; if the green colour is not produced quinidine is absent. Proceed E.

D. Detection of Cinchonine. The liquid after addition of KI is filtered and one drop water of ammonia added; set aside for a few minutes; there results:

1. Perfectly clear solution, in absence of cinchonine. Proceed E.

2. Turbid solution, if cinchonine is present. Proceed E.

E. Detection of Cinchonidine. If in the foregoing examination cinchonine or quinidine is found, the precipitate of tartrates (see A) is carefully washed with 15 to 20 c.c. Rochelle salt solution (1 to 20); were these alkaloids not found this washing is superfluous. The precipitate is dissolved off the filter by use of 3 c.c. dilute  $H_2SO_4$  (1 to 20); to the solution 2 c.c. ether and 1 c.c. water of ammonia are added, the mixture is well shaken for one minute and allowed to stand at rest for five minutes. This shaking and allowing to stand is repeated several times (the time allowed not to exceed a half-hour). Notice:

1. The ethereal layer and the sides of the test tube remain perfectly clear in absence of cinchonidine.

2. The ethereal layer and the sides of the test tube become cloudy, if cinchonidine is present.

Remarks referring to Quinine containing Quinidine. By this method one-fourth or one-half per cent. quinidine cannot be detected. Often 1 or 2 per cent. quinidine will give no reaction with KI, but the presence can be ascertained by the addition of water of ammonia, which is a more delicate test. In the examination of quinine with 10 per cent. quinidine nothing extraordinary is noticed, but in presence of 15 per cent. and more of this alkaloid it is noticed on addition of Rochelle salt that the tartrates are not precipitated; this does not, however, interfere with the further detection of quinidine. Should more than 15 per cent. quinidine be suspected, more KI must be used, otherwise it must be feared that the quinidine is not thoroughly precipitated and later may be mistaken for cinchonine. As a large percentage of quinidine prevents the separation of the tartrates and, as was found by special experiments, quinine sulphate mixed with quinidine sulphate is more difficultly soluble than the pure salt, there appears to exist a dependency between the two alkaloids.

Remarks referring to Quinine containing Cinchonidine. One-half per cent. of cinchonidine is easily detected within half an hour. Should the per cent. of cinchonidine be so minute that a precipitate cannot be clearly distinguished, absence of the alkaloid must be decided upon, as the reaction is so characteristic that there can

\* Excess of chlorine water prevents the thalleioquin reaction, while excess of ammonia favours it.

absolutely be no mistake. With 1 per cent. the reaction is more decided; with 2 per cent. there appears a deposit in the ether. Cinchonidine is recognised by the capillary rising of the precipitate beyond the ethereal layer, immediately after shaking the solution. Care must be exercised that not every slight turbidity at the line of contact of the two liquids be pronounced as cinchonidine; only in case the precipitate is capillary attracted by the side of the test tube can there be no doubt regarding its presence. If the quinine contains 10 per cent. cinchonidine there appears at the line of contact of the two liquids a white cretaceous ring. Cinchonidine, in the absence of quinine, is separated as a white precipitate in the ethereal layer.

Remarks referring to Quinine containing Cinchonine. One per cent. cinchonine is easily recognised; the more cinchonine present in the quinine the greater the precipitate with water of ammonia. When more than 5 per cent. cinchonine is present there forms on addition of KI a turbidity of cinchonine hydriodate, which can easily be mistaken for separated quinidine; 10 per cent. causes a precipitate of a tenacious character similar to quinidine hydriodate.

#### NOTES UPON METHODS FOR ESTIMATING THE QUANTITIES OF HOMOLOGOUS ACIDS PRESENT IN ARTIFICIAL SALICYLIC ACID.\*

By ERWIN E. EWELL AND ALBERT B. PRESCOTT.

Of the homologous phenols associated in the distillates of coal-tar the first member is the chief constituent of ordinary carbolic acid, but, it is well known, the higher members are not altogether absent. Considerable percentages of higher phenols are common in carbolic acid. Since salicylic acid is in greater part manufactured from carbolic acid by Kolbe's process,† inquiry naturally arises as to what becomes of the higher phenols of carbolic acid used in this manufacture. It had been found, some time ago,‡ that when the higher phenols of carbolic acid are treated as they are in Kolbe's method they are changed into homologues of salicylic acid; the cresols into hydroxy-toluic acids ( $C_7H_6.OH.CO_2H$ ), and the xylenols into hydroxy-xyleneic acids ( $C_8H_8.OH.CO_2H$ ) just as phenol proper is changed into a hydroxy-benzoic acid.§

The presence of some other acids besides true salicylic acid (ortho-hydroxy-benzoic acid) in the article of salicylic acid of commerce was reported by Mr. Williams in 1878.<sup>||</sup> He found the calcium salt of the foreign acid to be much more soluble in water than calcium salicylate is. By neutralising a hot aqueous solution of salicylic acid with calcium carbonate, causing the salicylate of calcium to crystallise out as completely as practicable, and then acidulating the mother liquor, the unknown acid was obtained. In certain physical characters this acid was found to be distinctly different from salicylic acid,

\* *Pharm. Record*.

† Kolbe, 1874; *Jour. prakt. Chem.* [2] 10, 89; *Jour. Chem. Soc.*, 28, 260; *Watts's Dict. Chem.*, 7, 1065; *Proc. Am. Phar. Assoc.*, 23, 374; 27, 463. Schmitt, 1885: *Jour. prakt. Chem.*, [2] 31, 397-411; *Jour. Chem. Soc.*, 48, 982; *Pharm. Jour. Trans.*, [3] 5, 421.

‡ Biederman and Pike, 1872: *Ber. d. Chem. Ges.*, 5, 323; *Watts's Dict.*, 7, 394, 8, 2023.

§ The ortho-compound,  $C_6H_4.OH.CO_2H$  ( $OH : CO_2H = 1 : 2$ ). The isomers and their homologues are given in Prescott's "Organic Analysis," pp. 394, 434, 443.

|| J. Williams, 1878: *Phar. Jour. Trans.* [3] 8, 785; *Proc. Am. Phar. Assoc.*, 26, 536.



and from the other two hydroxy-benzoic acids. In the leading chemical relations, however, the unknown acid was found to agree with salicylic acid and the isomers of the latter. The acid liberated from the crystals of calcium salt, after purification, was found to agree in all points with true salicylic acid. Mr. Williams concluded that the acid not salicylic formed fifteen to twenty-five per cent. of the better grades of salicylic acid of the market. Very little further investigation of the presence and proportion of the homologous acids has been reported. In 1883 Dr. E. R. Squibb\* gave the opinion that "the better grades of the well-crystallised acid of the market contain four to five per cent. of something which is not salicylic acid," and for which, he says, he knows no test.

The physiological and therapeutic value of the homologous acids in medicinal salicylic acid are certainly deserving of careful study, in view of the large doses in which the agent is used, and the somewhat variable effects reported of these doses.

#### 1.—A Method by Acidimetry.

The molecular weights of the homologous acids show the following differences, those of  $\text{CH}_2 = 13.97$ , in arithmetical increase.

Salicylic acid (hydroxy-benzoic acids),  $\text{C}_6\text{H}_4\text{OH.CO}_2\text{H} = 137.67$ .

Hydroxy-toluic acids,  $\text{C}_7\text{H}_6\text{OH.CO}_2\text{H} = 151.64$ .

Hydroxy-xyleneic acids,  $\text{C}_8\text{H}_6\text{OH.CO}_2\text{H} = 165.61$

And of hundredth normal solution of alkali ( $\frac{N}{100}$ ) needed to neutralise these acids and form monobasic salts :

1 gram. of salicylic acid requires 726.3 c.cm.

1 gram. of a hydroxy-toluic acid requires 659.4 c.cm.

1 gram. of a hydroxy-xyleneic acid requires 603.8 c.cm.

In the application of this method it was assumed that hydroxy-toluic acids,  $\text{C}_8\text{H}_6\text{O}_3 = 151.64$ , fairly represent the total acids of molecular weight above that of salicylic acid. This can be justified both because there can be only a very small quantity of xlenols in carbolic acid of respectable quantity, and because a given percentage of the xlenol product would indicate a larger percentage of the less objectionable cresol product. Thus, of the ( $\frac{N}{100}$ ) alkali solution to neutralise 1 gram. of the acid,

Hydroxy-toluic acids take 66.9 c.cm. less than salicylic

Hydroxy-xyleneic acids take 122.5 c.cm. less than salicylic.

One per cent. of a hydroxy-xyleneic acid would indicate 1.8 per cent. of a hydroxy-toluic acid. Then the saturating capacity of articles of "salicylic acid" made from carbolic acid will stand as follows :—

#### FOR ONE GRM. OF THE ACID TESTED.

Salicylic acid, absolute			726.3	c.cm.	$\frac{N}{100}$	alkali.
"	"	with 5 per cent. hydroxy-toluic	723.0	"	"	"
"	"	" 10 "	719.6	"	"	"
"	"	" 15 "	716.3	"	"	"
"	"	" 20 "	712.4	"	"	"
"	"	" 25 "	709.5	"	"	"

\* *The Ephemeris*, 1, 411, Nov., 1883.

In making trial of the calculated saturating powers of salicylic acid and its homologues, potassa and soda were found to work equally well in the standard solutions ( $\frac{N}{100}$ ), and phenol-phthalein proved thoroughly satisfactory as an indicator of the end-reaction, while litmus is wholly incapable of being used in this estimation. The titrations are to be made as follows:—

About  $\frac{1}{2}$  grm. of the acid to be tested, dried at or below  $65^{\circ}$  C. to constant weight, is accurately weighed, placed in a beaker of about  $\frac{1}{2}$  litre's capacity, some drops of the (alcoholic) solution of phenol-phthalein added, and (without adding water for solution) the hundredth-normal solution of alkali is run in from the burette, while stirring, until the end-reaction is approached. The beaker is now placed upon wire cloth over the flame (or otherwise promptly heated) while gently stirred, only long enough to complete the solution of the acid, and without decided boiling, when the heat is removed, the beaker-sides rinsed down with a little distilled water, and the titration completed. The weight taken : 1,000 :: c.cm. required :  $x$  = c.cm. for 1 grm., to be compared with the table above.

In drying salicylic acid for a constant weight it was found that this could be attained at  $65^{\circ}$  C., while at  $70^{\circ}$  to  $75^{\circ}$  C. there was a constant loss, in a short time sufficient to be registered by the balance. A portion of the purified acid from oil of wintergreen, placed between watch-glasses, arranged for sublimation, and heated to  $115^{\circ}$  C., promptly gave a sublimate of colourless crystals of good size. In aqueous solution, it suffers loss by boiling at common air pressure.

Acidimetry under the directions given above was tried upon a sample of purified acid from oil of wintergreen, a sample prepared for the purpose, in the way followed by Williams:\* one ounce of oil of wintergreen was saponified by potassium hydrate solution, the liquid cooled and acidulated with hydrochloric acid, the precipitate four times crystallised from boiling water, the hot solution filtered through strictly purified animal charcoal and re-crystallised, and lastly twice re-crystallised from alcohol. The average of several titrations of this purified salicylic acid agreed very closely with the calculated quantity, 726.3 c.cm. ( $\frac{N}{100}$ ) alkali for 1 grm. of the acid.

Applying the same volumetric reagents in the same way to a sample of salicylic acid of the ordinary market,† in four titrations an average of 714.3 c.cm. was obtained—corresponding to a proportion of 15 to 20 per cent. of homologous acids calculated as a hydroxy-toluic acid.

With titrations of good ordinary care, using verified instruments of such delicacy as every chemist requires for analytical work, acidimetry can reveal quantities of hydroxy-toluic acids as low as 4 or 5 per cent., other interfering impurities being absent.

(To be continued.)

## ON THE RELATIVE VALUE OF DIFFERENT PEPSIN TESTS.

BY JAMES H. STEBBINS, JR.

(Continued from page 198.)

Lately my attention has been called to a new pepsin test, which I will designate by its author's name, the "Manwaring test." In this test Manwaring has tried to avoid as much as possible the bad points of the U. S. P. test; but in doing this he has stumbled against other sources of error which I will try to make clear farther on.

\* *Phar. Jour. Trans.* [2], 8, 785.

† In appearance the sample was an indistinctly crystalline powder of a slightly pinkish colour.



The test can best be described in the words of its author:—

"The design of the following mode of testing the dissolving power of pepsin is to conform as nearly as possible to the U. S. P. test, which, contemplating the testing of the saccharated form, makes no provision for the proportion of acidulated water to be used with a pure pepsin.

"On the basis that 1 part of a pure pepsin is capable of dissolving 1,000 times its weight of coagulated egg albumen in six hours, a saccharated pepsin made with a pure pepsin of U. S. P. strength would contain 5 per cent. of pure pepsin; therefore if 1 grain of a U. S. P. *saccharated* pepsin is to be tested in the presence of 500 grains of acidulated water, then 1 grain of a pure pepsin should be tested in the presence of 10,000 grains acidulated water, to equal the same proportion of water and acid used for the *actual* quantity of pure pepsin contained in a U. S. P. *saccharated* pepsin when tested according to the U. S. P."

In order to render the weighing of small quantities of pure pepsin as easy as possible to the pharmacist, Manwaring recommends that it should be saccharated, and for this purpose he gives the following recipe:—

R. Saccharated pepsin, consisting of—

Pure pepsin	..	..	..	..	..	..	..	1	gram.
Milk-sugar	..	..	..	..	..	..	..	19	"

To make the test, take of the above saccharated pepsin 0.3 gram. (= 0.015 gram pure pepsin)—

Coagulated egg albumen	..	..	..	..	..	22.5	grms.
Acidulated water, consisting of—							
Distilled water, 100 c.c.							
Hydrochloric acid, U. S. P., 1.25						154	c.c.

The eggs are to be boiled for fifteen minutes, and the whites pressed (by means of a spatula) through a (preferably flat) 30-mesh sieve. For the sake of uniformity, the egg whites should be cut into small pieces and thoroughly mixed before being passed through the sieve.

The mixture should be maintained at 100-105° F. for six hours, and agitated thoroughly about every half-hour.

At the end of six hours the temperature of the bath should be quickly run up above 150° F. to destroy the pepsin, then the bath with contained bottles allowed to remain undisturbed over night, that the undissolved albumen may settle.

If the test bottle has been kept securely corked during the test, or if by previously weighing bottle and contents and afterwards making up with water any loss from evaporation, the quantity of albumen dissolved may be easily determined as follows:

From the settled contents of the test bottle pipette off 10 c.c. and evaporate to dryness—until weight is constant—in a watch glass. From this dry residue figure as follows (1 pt. of peptone or intermediate products representing 1 pt. of original albumen):

Suppose 10 c.c. of the liquid = 0.2 gram. dry residue;  $\frac{1}{1.1}$  times its weight = the quantity of water contained in the 10 c.c. that was derived from the albumen dissolved; 0 c.c. of liquid less 1.4 c.c. of water leaves 8.6 c.c. water taken from the 154 c.c. of acidulated water in making the test.

1.6 grms. or 8 times 0.2 grm: dry residue = the quantity of albumen in its natural state as originally used, that has been dissolved in the 10 c.c. of liquid evaporated to dryness.

Therefore, if 8.6 c.c. acidulated water holds 1.6 grm. egg-albumen,

then 154 c.c.   "   "   "   28.5   "   "

Then, as 0.015 grm. pepsin dissolved 28.5 grms. coagulated egg-albumen, 1 pint would dissolve 1900 times its weight.

The use of the multiplier 7 and 8 is based on the fact that egg-albumen averages 12 per cent., or  $\frac{1}{8}$  dry.

As will be seen, this test is quite a departure from the U. S. P. test, and, in some respects, is an improvement upon the latter, but I object to it on several points, viz.:

1. It makes no provision for other than concentrated, or, as Manwaring calls them, *pure* pepsins, while in reality the number of these is small compared to the saccharated pepsins.

A person desiring to essay a saccharated pepsin by this test would be at an utter loss to know how much pepsin should be weighed out, or how much acidulated water should be employed, unless he knew that the pepsin under examination was of U. S. P. strength.

This cannot be ascertained without submitting the sample to a complete chemical analysis, and hence a great deal of trouble and erroneous results are apt to ensue.

I have encountered this very trouble myself, as may be seen from the following figures:

A sample of pepsin, purchased as a pure pepsin (pepsin C in the tables), but which in reality was a saccharated pepsin, gave by this method 420, i.e., it is supposed to dissolve 420 times its weight of coagulated albumen.

Not knowing the strength of this saccharated article in pepsin, I was at a loss to know how much should be used in the test, and therefore decided to look upon it as a concentrated pepsin. In consequence of this decision on my part the pepsin was saccharated according to M.'s directions, and 0.3 grm. of this resaccharated pepsin used in the test, with the above result. The result obtained undoubtedly does the pepsin injustice, for it is probable that if I had not resaccharated the original article, and consequently diluted it to a much greater extent, that a much higher test would have been recorded.

The same applies also to the following pepsins, which were in the saccharated condition when bought and were resaccharated by me before testing.

Brand of Pepsin.							Grs. Albumen dissolved in six hours.
Pepsin A..	..	..	..	..	..	..	550.6
" B..	..	..	..	..	..	..	516.6
" C..	..	..	..	..	..	..	420.
" D..	..	..	..	..	..	..	398.6

(To be Continued.)



## ON SOURCES OF ERROR IN DETERMINATIONS OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. ATWATER.

*(Continued from page 196.)*

The experiments cited by Mr. Ball and myself in the previous article of this series bear striking testimony to the danger of loss if there is not sufficient contact between the gaseous distillation products and the heated soda-lime. In determinations of casein by our usual method (which involves close packing of the soda-lime in the tube, the use of coarse particles of soda-lime for the anterior layer, heating the anterior layer before the distillation products begin to be developed, and keeping it well heated until the combustion is finished), the whole of the nitrogen, 12.43 per cent., was obtained as ammonia. But when a channel was left in the tube (and fine soda-lime was used for the anterior layer), so that there was less contact between distillation products and heated soda-lime, the nitrogen obtained as ammonia amounted to only from 12.25 to 11.65 per cent. of the weight of the casein instead of 12.43 per cent.; that is to say, from 1.4 to 1.4 per cent. (on the average 3.3 per cent.) of the whole nitrogen escaped ammonification. Then, again with the channel, the anterior layer was made shorter, 7.5 cm. instead of 12 cm., the nitrogen obtained as ammonia fell as low as 10.51 per cent., the rest, 15.4 per cent. of the whole, escaping ammonification. The average loss under these last conditions was 6.4 per cent. of the whole nitrogen. Even more striking illustrations of the failure of distillation products to be changed to ammonia are given in the experiments with strychnine described in the same article. In these but a fraction, at times very small, of the total nitrogen was obtained as ammonia, although especial effort was made to secure complete ammonification.

But little consideration of the matter is needed to show that the formation of volatile nitrogenous products which would escape ammonification by the soda-lime and detection in the acid is a very natural occurrence.

The protein compounds when subjected to destructive distillation yield a great variety of products, among which are, besides water, carbon dioxide, hydrogen sulphide, and hydrocarbons, a great variety of volatile nitrogenous compounds, including ammonia, ammonia salts of the fatty acids, amines derived from the paraffins, members of the aniline and pyridine series, pyrrol, etc., etc.\* By heating in the presence of alkalies and other reagents a great variety of other compounds, with the rest a number of acids of the fatty acid series and amido acids belonging to this and the aromatic series, among which latter are leucine and tyrosine. By the action of enzymes and microbes, products akin to the alkaloids are formed. The alkaloids break up into compounds such as were mentioned above, and numerous others, including members of the quinoline series.

Now these nitrogenous products vary greatly in respect to their volatility and the ease with which they are changed to ammonia when heated in presence of soda-lime (*i.e.*, of water vapour at high temperature). The specific data at hand regarding the ease with which they are ammonified are meagre. There are, however, observations bearing upon this point. For instance, Goldberg finds that some of the more complex azo-

\* See Handw. d. Chem. 2, 1161-1173, and E. Salkowski, Ztschr. anal. Chem. 16, 1877, 261 and 408.

compounds, with soda-lime, yield all their nitrogen as ammonia, while the simpler ones fail to do so, part escaping.\* There is nothing more natural than that in soda-lime combustions such volatile nitrogenous products should be formed and escape ammonification.

A fact of interest here is that some, at least, of the compounds which are most difficult to determine by soda-lime are the ones that are most readily broken up into very volatile products. Such are guanidine, kynurenic acid which may yield quinoline, leucine, and quinine and other alkaloids. It is also worth noting that some of the alkaloids, the nitrogen of which it is so difficult to transform entirely into ammonia by soda-lime, manifest a similar behaviour in treatment with sulphuric acid and other reagents by Kjeldahl's method,† and perhaps for similar reasons.

In looking over the literature of the subject, which is quite extensive, one cannot help being impressed by the fact that certain nitrogenous compounds very frequently, and others almost uniformly, fail to yield all of their nitrogen in the form of ammonia when heated with soda-lime. Whatever may be the differences of manipulation of different experimenters, nearly all find it impossible to get all of their nitrogen in that form from such substances as leucine and some of the alkaloids. But in some cases, as for instance, those of Märcker and Abesser above quoted, the nitrogen has been obtained by precipitating platinic chloride even when it was not obtained by titration. Leucine‡ is, in this respect, an especially refractory compound, though chitin,§ gluten-protein,|| and, in some instances, casein¶ (milk) and peptones\*\* have given trouble in this way.

The interesting researches of Schützenberger†† have indicated that the protein compounds (albuminoids and gelatinoids) are made up of a variety of simpler compounds, among which leucine and another, or perhaps several other compounds closely allied to it, are among the important constituents. That is to say, under the influence of various agencies the protein compounds split up into a large variety of simpler compounds, among which leucine and its congeners play an important part. That these latter might be easily formed from protein compounds in the breaking up which occurs in the ordinary heating with soda-lime is a very natural inference from the facts at hand. But from its constitution, leucine might be expected to readily be split up into volatile nitrogenous compounds. Such observations as those of Märcker and Strecker, above quoted, would seem to give reasonable assurance that it actually does so. In view of these facts it will be easy to assume that in ordinary

\* Ber. d. chem. Ges. 25, 46.

† Ztschr. anal. Chem. 22, 1883, 379.

‡ E.g. Märcker and Abesser, loc. cit.; Ritthausen and Kreussler, J. prakt. Chem. 111, 1871; Ritthausen, *ibid.* 116, 1874, 17.

§ Bütschli, Ztschr. anal. Chem. 16, 1877, 409.

|| Märcker and Abesser, loc. cit.; Kreussler, Landw. Vs. St. 31, 1884, 248. See also Ritthausen, loc. cit.

¶ Lehmann, Ztschr. anal. Chem. 15, 1876, 113; Musso, *ibid.* 16, 1877, 413; Menozzi, *Jab. Agr. Chem.* 21, 1878, 474; Kreussler, loc. cit.

\*\* Gruber and Feder, Ztschr. f. Biol. 16, 1880, 381.

†† Bull. Soc. Chim. 23-30, 1875-1878, and *Chimie Générale* 131, 407.



combustions by soda-lime there is a tendency to the formation of nitrogenous distillation products, such as come from leucine and analogous compounds, and which would escape ammonification.† Such being the case, it is easy to see how volatile nitrogenous compounds might often escape ammonification unless the greatest pains were taken to insure the most perfect contact between them and the heated soda-lime (heated water vapour) and that sometimes the ammonification might be incomplete despite the greatest care. If this be correct, the difficulty of getting all of the nitrogen of peptones into form of ammonia would accord with the fact that the peptones are products of change of the albuminoids, which change, carried further, results in the cleavage of the latter into leucine and other allied products. It would also be easy to theorise regarding the constitution of the alkali albumins and casein, and to imagine that they might likewise be on the way toward the process of cleavage by which leucine and its decomposition products are produced. Of course this is speculation, but it at least helps us to see how the observed facts might occur; and irrespective of any such hypotheses, the known facts are sufficient to warrant the assumption that under the influence of high heat in the presence of soda-lime, numerous volatile nitrogenous compounds might be expected to be formed, some of which would resist ammonification; and that certain classes of protein compounds would be especially prone to such decomposition, even though, in the present state of our knowledge, we are unable to say just exactly what those compounds are, or what are the processes of cleavage they go through, or what are the nitrogenous products that resist ammonification. An illustration of the difficulty of ammonifying some of these volatile compounds is found in the above-mentioned case of strychnine in the experiments detailed in the previous article of this series. Neither the most pains taking effort to secure contact between the gases and the soda-lime by the Will-Varrentrapp method, nor by the greatest care in heating by that of Kjeldahl, sufficed to convert all the nitrogen into ammonia. On the other hand, the results with casein, when means were taken to secure adequate contact with soda-lime and to avoid dissociation, were most satisfactory. But when a channel was left in the tube so that the distillation products were not brought into close contact with the soda-lime, the loss was at times very large.

The lesson which all these considerations teach, and which is enforced by those cited in the previous articles, is the importance of providing intimate and sufficient contact between the distillation products and the heated soda-lime. This is done by fine pulverization and intimate mixture of substance with soda-lime; by having the tube closely packed with soda-lime to avoid open spaces; by providing a long enough anterior layer of soda-lime, and by heating this layer well before the gases are disengaged, and keeping it well heated until the combustion is finished. At least such seems to me the just inference from the facts at hand.

† See also Hofmann, Ann. Chem. (Liebig) 79, 29; Wertheim, Jour. prakt. Chem., 53, 431; Williams, loc. cit. 76, 333; and Michael, this Journal 7, 182, for decompositions of alkaloids and other compounds, bearing upon this question.

(To be continued.)

## MONTHLY RECORD OF GENERAL RESEARCHES INTO ANALYTICAL CHEMISTRY.

REMOVAL OF FUSED MASSES FROM PLATINUM CRUCIBLES. L. L. DE KONINCK. *Zeitschr. f. angew. Chemie*, No. 20.—When fusions are performed in large dishes, no particular trouble is experienced in dissolving the fused mass; but if done in deep crucibles it is better to first empty them. Many devices have been proposed, but they generally only succeed with new crucibles, and are apt to spoil them in the long run. The author now operates as follows:—As soon as the fusion is completed, the coiled end of a platinum wire is put into the mass, which is then allowed to cool. The wire is about 10 cm. long, and ends in a loop, so that it may be suspended from a hook. When suspended, the crucible is brought at about half an inch distance from a pipeclay triangle, and then suddenly heated. In a few seconds the crucible will detach itself and drop into the triangle, whilst its contents stick to the wire. The small quantity of salts still adhering to the crucible may, of course, be removed by suitable means. The wire is now made to slip into a beaker filled with water or acid, when, owing to the salts being on the top of the fluid, solution takes place with great rapidity. L. DE K.

TESTS FOR CARBOHYDRATES. L. V. UDRANSZKY, *Zeitschr. f. Phys. Chem.*, May, 1888.—Undoubtedly the furfural reactions furnish the most delicate tests for the carbohydrates. H. Schiff uses a test paper made by immersing paper in a mixture of equal volumes of xylidin and glacial acetic acid diluted with alcohol and drying. A small quantity of the substance to be tested is heated with a slight excess of concentrated sulphuric acid and the test paper held in the evolved vapours, a beautiful red colour is produced owing to the formation of the furoxylidin. It will detect as little as 0.00007 gm. glucose in an aqueous solution. The author uses a furfural reaction, even more delicate than the above, detecting 0.000028 gm. glucose in solution. One drop of a dilute solution to be tested is mixed with two drops of a 15 per cent. alcoholic solution of  $\alpha$ -naphthol in a test tube and  $\frac{1}{2}$  c.c. concentrated sulphuric acid is carefully poured in to form a distinct layer. If at the line of contact a violet colour above a green layer is produced, carbohydrates are present. Urine is diluted with 9 volumes of water and one drop proceeded with as above. If the violet colour is not produced, the urine is considered normal; if the colour is produced, the urine may be considered abnormal because it yields a quantity of furfural which is also obtained from a glucose solution containing at least 0.5 per cent. By means of these two tests carbohydrates were detected in all urines examined: albumen perfectly free from carbohydrates heated with concentrated acids formed furfural which was recognised in the distillates, establishing for the first time by chemical reactions a close relationship between the albuminoids and the carbohydrates. In testing urine for carbohydrates, if albumen be present in larger quantities it must first be removed, small quantities do not introduce appreciable errors, owing to the small quantity of urine taken. Fehling's solution under the most favourable conditions failed to detect less than 0.00012 gm. glucose in aqueous solution; testing urine by the three tests the bodies other than carbohydrates decrease the delicacy of Fehling's test to a greater degree than the first two tests. W. H. D.



**DELICATE TESTS FOR AROMATIC AMINES.** A. THL. *Chem. Zeitung*, 76.—The oxidation of aromatic amines yields complicated condensation products, which are nearly all splendid dyes. The author tried manganic dioxide, but in presence of mineral acids no striking result was obtained. Very interesting results were, however, got by using hydrated manganic dioxide, in conjunction with acetic, oxalic or tartaric acids. Toluidin oxalate gives with the manganic dioxide a red colour, changing to green on heating, but turning red again on boiling. At the same time a black colouring matter precipitates, which dissolves in spirit with a red colour. Xylidin acetate similarly treated gives a splendid reddish-violet, which gradually darkens. Dimethylanilin oxalate, diethylanilin acetate, in fact all aromatic amines, give the most splendid display of colours. These colours may also be prepared by making the amine salts with manganic dioxide to a paste, and heating until the mass looks yellowish-red. The colouring matters may then be extracted with water or spirit. If a thread of wool is boiled in a very weak solution of toluidin or xylidin oxalate with addition of some manganic dioxide, it becomes black, but after washing, it will look of a nice dark-brown colour. Cotton wool is not dyed under the circumstances. If the solution of toluidin oxalate is first boiled for some time with manganic dioxide, and then allowed to settle, it will colour a piece of wool beautiful reddish-violet. The other amines will also colour the wool in various shades. The colours seem to stand pretty well the action of light and soap.

L. DE K.

**CONVENIENT PREPARATION OF CHLORINE FOR ANALYSES.** L.L. DE KONINCK. *Zeitschr. f. angew. Chemie*, No. 18.—The author for several years advocated the use of a Kipp's apparatus, which after being charged with manganic dioxide and hydrochloric acid had to be heated on a water bath. Owing to the many breakages, the author has now constructed an apparatus by means of which the chlorine may be prepared in the cold. Hydrochloric acid is evolved in the author's apparatus (see *Zeitschr. f. angew. Chemie*, page 353) and passed through a tower, containing granulated manganic dioxide, when the usual reaction takes place, and chlorine is liberated. The gas is then dried by means of calcium chloride or sulphuric acid. For the preparation of small quantities, the author used a Peligot's tube (a U-tube with three bulbs), one side of which is filled with lumps of manganese of the size of a pea, and the other with calcium chloride. For large quantities the author prefers R. Muencke's drying cylinder.

L. DE K.

**DETECTION OF MERCURY IN URINE.** DR. HIELBIG. *Pharm. Zeit. Russl.*—The author adds to 100 c.c. of urine 10 c.c. of diluted hydrochloric acid (1 to 2) and 2 to 3 grains of recent copper scrapings, and evaporates in a porcelain dish to about 5 c.c., stirring occasionally. The mercury, if present, will precipitate upon the copper, which is taken out, rinsed with water, boiling alcohol and ether, allowing it to dry at ordinary temperature. The copper is then heated in a glass tube of 5 mm. diameter for 2 or 3 minutes; the mercury sublimes. After cooling, the copper is shaken out, a few fragments of iodine introduced, and the tube heated gently, when the vapours of iodine will combine with the mercury forming the biniodide. Experience has shown that the above

proportions are the best. The test is very sensitive. Examination of the tube by the microscope is an aid to the detection of very minute quantities. W. H. D.

**TESTING AMBER VARNISH.** W. SONNE. *Zeitschr. f. angew. Chemie*, No. 18.—Commercial amber varnish is made by dissolving amber or colophony-amber in linseed oil, varnish, and turps. In many cases it is made without the expensive amber, and an analyst is sometimes asked his opinion, whether a sample is genuine, viz., really made with amber. The best way is to try for succinic acid, although even a genuine article only contains small quantities of this substance, as a large quantity volatilises during the heating of the varnish. The detection is, however, difficult, owing to the nature of the article. Neither boiling with hydrochloric acid, nor treatment with alcoholic potash, extracts any succinic acid. The author's plan is to treat the sample with nitric acid of 1.20 specific gravity.\* He proceeds as follows:—20 grms. of the varnish are put into a flask of about 300 c. c. capacity, and heated on a sand bath with 50 c. c. of the nitric acid. When action sets in, the flask must be somewhat cooled to prevent a too fierce oxidation, when it may be again gently heated for about fifteen minutes. The acid, which holds all succinic acid in solution, is now poured off and the insoluble resinous mass washed with water. The acid is evaporated in the water bath, a little water being from time to time added. When the acid has been completely expelled, the remaining syrup is dissolved in about 10 c. c. of water, and this solution shaken with 100 c. c. of ether. After distilling off the ether, the residue is put in a watch glass and put under a desiccator. After about twelve hours, crystals of succinic acid separate out and the amount gradually increases. The mother liquor being removed by means of blotting paper, the crystals may now be tried by the usual tests for succinic acid. It is thus possible to answer within twenty-four hours the question whether a sample of amber varnish is really deserving of the name. L. DE K.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

**FUSEL OIL IN SPIRITS.**—E. SELL.—*Zeitschr. f. angew. Chemie*, No. 20.—Among the best processes known is the method of Röse, an elaborate abstract of which will be found in the *Analyst*, vol xi. It is, however, necessary to strictly adhere to the various precautions, otherwise the results will be faulty. The author also studied the influence of ethereal oils, and found them not to seriously interfere with the accuracy of the process. They may (if present in larger quantity) be practically got rid of by rectifying the spirit with caustic potash.

200 c. c. of the sample are made strongly alkaline with caustic potash and put into a retort containing a few lumps of pumice-stone (to prevent bumping) and the retort is then connected with a Liebig's condenser. The receiver consists of a narrow graduated cylinder, and 4.5ths of the brandy are distilled off. The distillate is now made up to

\* Note by abstractor. May not some succinic acid be actually produced by oxidation of fatty matter?—L. DE K.



original volume. After diluting or strengthening, if the percentage of alcohol should not be exactly 30 by volume, it is ready for the treatment with chloroform.

L. DE K.

**BORACIC ACID AS A PRESERVATIVE.** EMMERICH. *Chem. Zeitung*, No. 76.—Boracic acid only acts when present in large quantity. It prevents the growth and multiplication of germs, but does not kill them even in a 1 per cent. solution. Experiments with milk gave very unsatisfactory results, as an addition of 4 per cent. boracic acid only preserved the milk for four days. Horseflesh may be preserved for six weeks by the use of 3 per cent. of the acid. Boracic acid is supposed to be harmless, but recent investigators, including the author, prove it to be dangerous, as it strongly acts upon the mucous membrane of the large intestine. A dose of 4 grms. killed a large rabbit; 2 grms. made a dog very sick.

The acid is much used in Sweden for preserving fish and milk, but cases of poisoning have already occurred in that country. Long continued use of the acid is not favourable to good health, and at all events its addition to milk should be prohibited.

L. DE K.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

**DISTINGUISHING CITRIC FROM TARTARIC AND MALIC ACIDS.** PROFESSOR MEAU. *Zeit. fur. Anal. Chemie.*—The author heats the acid with 0.7 per cent. of glycerin until vapours of acrolein are evolved; the mass is then taken up by a little ammonia, the greater part of which is afterwards expelled by heating. A few drops of nitric acid (the fuming acid diluted with five parts of water) are then added. Under this treatment citric acid yields a green compound, which turns blue on heating. Tartaric and malic acids do not produce the same coloration.

W. H. D.

#### REVIEWS.

**BOILER WATERS.** BY WALTER LEE BROWN. Chicago: Shepard and Co.

THIS is a record of the author's work on the waters available for locomotive purposes along the whole length of the Chicago, Burlington, and Quincy Railway. Two hundred and fourteen samples have been analysed, classified, and recorded. The author divides the total solids as follows:—(a) The *incrusting solids* in grains per gallon representing the sum of the following, viz., carbonates of lime and magnesia-sulphates of the same bases, silica, alumina and oxide, (or carbonate) of iron. The author admits that magnesium sulphate may not be universally viewed as a true "scale former," but he prefers to include it as such for various reasons, and thus get truly comparable results in all the waters. (b) The *non-incrusting solids* in grains per gallon which are obtained by deducting the incrusting solids from the total residue, and are also termed the *corroding* or *sludge-producing* solids. They are manifestly alkaline salts, together with any organic matters present. The author adopts the following scale of comparative rating:—

Less than 8 grains of <i>incrusting solids</i> per gallon, considered as Very good.				
8 to 15	"	"	"	Good.
15 to 20	"	"	"	Fair.
20 to 30	"	"	"	Poor.
30 to 40	"	"	"	Bad.
Over 40	"	"	"	Very bad.

These are practically the standards adopted by the American Association of Railway Chemists, except in the case of the first degree of "very good." The Association also determined that the best practical form of report was to state the incrusting solids in lbs. per 1,000 gallons. It would be well if English analysts were to agree upon similar forms and standards, which are decidedly more comprehensible to steam users in general.

There is only one original process given in the book, and here we fear that we British chemists will not go along with our American *confreres*. It is true he concludes it by the statement that "the above process is not claimed to give the utmost scientific accuracy, but it is much better than any 'hardness' method." We have not space to give the details, but will only remark that a process providing for the separation of lime and magnesia in pure aqueous solution, by ammonia and ammonium oxalate, without any previous addition of ammonium chloride, and finally requiring the addition of one quarter to the amount found (to compensate for the want of solubility of calcium sulphate, is in our opinion very far short of scientific accuracy indeed.

THE ANALYST'S LABORATORY COMPANION. BY ALFRED E. JOHNSON. London: J. and A. Churchill.

THIS is a table book of 90 pages, including all the usual tables required by analysts, with a set of 7 figure logarithms from 1 to 1,000. The printing is clear and distinct, and the book, so far as can be seen from a perusal, seems to be very free from printer's errors. There are a few original tables given, which will be found useful in the estimation of carbohydrates with the polariscope, and in the estimation of chicory in coffee, etc. There is also a very good table for calculation of phosphates specially worked out for agricultural analysts.

#### LAW NOTES.

SELLING COTTON-SEED OIL FOR OLIVE OIL.—Mr. Alfred Fresson, chemist and druggist, Queen Street, Burslem, was on the 16th ult., charged before the Staffordshire Potteries Stipendiary, at the instance of Mr. E. W. H. Knight, inspector under the Food and Drugs Act, with having sold as olive oil an article that the County Analyst, (Mr. Jones, Wolverhampton) declared was entirely cotton-seed oil. Mr. Knight said that the defendant sold in a bottle labelled "fine olive oil" an article which upon being analysed turned out to be an inferior vegetable oil. As he was a chemist he ought to know what he was selling, and not impose on his customers at a cost of tenpence, an article which was worth about threepence. Cases of this nature were constantly cropping up, and he therefore asked the magistrates to inflict an exemplary penalty, so as to put a stop to the practice.

The defence was that the accused purchased the oil in question from the wholesale house as a second quality of olive oil, and the price he paid for it was 3s. 6d., per gallon. Mr. Knight said 3s. 6d., per gallon was a very low price for olive oil. Tradesmen who desired to supply their customers with a pure article would give a fair and reasonable price for it. The article sold by the defendant was cotton-seed oil, which was only worth about 1s. 10d., per gallon. The accused was legally liable for the adulteration, but if he had acted innocently through being misled by the wholesale dealers, he could recover from them any penalty that might be imposed upon him. The Bench said they looked upon the case as a very serious one. Persons going to a chemist's shop especially ought to be supplied with pure articles, as their purchases were very frequently required for medicinal purposes. The defendant would have to pay a fine of £5, together with 19s. costs.

SALE OF ADULTERATED LARD.—At the Liverpool Police Court on the 20th ult., before the stipendiary magistrate, a grocer named Hugh O'Neil, carrying on business in Brick Road, was charged with selling adulterated lard without a label. Inspector Baker stated that when he bought a sample of the lard, consisting of 2 lbs., he was told by the assistant in the shop that it was not pure, but it had no label whatever upon it.

Defendant stated that the inspector refused to take a sample of the pure lard, and he was told what he took was not lard. It was a material which was sold to bakers, who preferred it to pure lard for their purpose. Defendant had since ceased to sell it.

Mr. Raffles reminded the defendant that the offence was in not labelling the article, and he imposed a fine of 5s. and costs.



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## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE above Society resumed its meetings on the 14th ult., when an ordinary meeting was held at Burlington House, Piccadilly, Vice-President Dr. Muter being in the chair.

The minutes of the June London meeting and the Country, meeting at Sheffield were read and confirmed.

The following gentlemen were proposed for election :

As member—R. W. Woosnam, analyst to the Dairy Supply Company. As associate—A. J. Stoney, assistant to Mr. L. Briant.

On the ballot papers being opened, it was announced that the following gentlemen had been elected as members :—

Christopher Rawson, F.I.C., F.C.S., analytical chemist, Bradford ; Dr. E. B. Trueman, public analyst, Nottingham.

The following papers were read and discussed :—

"Determination of the Melting Points of Soap-Thickened Oils." By W. F. K. Stock.

"On Condensed Milk." By J. C. Shenstone.

"On the Preservation of Milk Samples." By H. D. Richmond.

The paper announced to be read by Dr. Dupré on a recent river pollution case was postponed.

The next meeting of the Society will be held at Burlington House on Wednesday, the 12th inst., at 8 o'clock.

The papers by Messrs. Stock and Richmond will be published next month.

## NOTES UPON THE ANALYSIS OF CONDENSED MILK.

By J. C. SHENSTONE.

*(Read at Meeting November, 1888.)*

I HAVE recently had occasion to analyse a large number of samples of condensed milk, and I venture to place before the Society an account of some experiments which appear to me of interest.

In order to estimate the commercial value of condensed milk the following data are necessary :—Total Solids, and by inference water, Fat, *Milk-Sugar*, *Cane-Sugar*, combined proteids and ash.

On opening a tin of condensed milk I pass a portion of its contents several times through a sieve of wire gauze, in order to separate any clots, and to secure uniformity of composition, and I preserve it in a well-closed bottle. No time is saved by making a solution and using separate portions, for owing to the viscosity of the product, each separate portion has still to be weighed to secure accurate results; moreover, such a solution soon coagulates.

I estimate total solids by diluting 0.5 grammes of condensed milk with about 5 c.c. of water, and after mixing them, dry by the water and air bath to a constant weight, but the solids of condensed milk rich in fat appear to yield the last traces of moisture even more reluctantly than natural milk, and, therefore, it is quite necessary to adopt all the precautions usually adopted in drying natural milk.

For estimating fat the paper coil method gives very satisfactory results. Two grammes of condensed milk may be diluted with about three grammes of water for this purpose.

Ritthausen's method for estimating the combined proteids in milk gives very satisfactory results in analysing condensed milk. I dilute 2 grammes of condensed milk with about 400 c.c. of boiling water, precipitate the proteids and the fat by adding 3 c.c. of solution of sulphate of copper, then add sufficient solution of potash to nearly, but not quite, neutralise the liberated acid. I collect and thoroughly wash the precipitate upon a weighed filter paper, dry, extract the fat, dry to a constant weight, and weigh; lastly reduce to an ash, and correct by deducting the weight of the filter paper and of the ash, this latter corrected for the calculated weight of the ash of the filter paper. I find the most convenient method for extracting the fat from the proteids is by means of ether with a Soxhlet tube; a thread can be attached to the filter paper with which to withdraw it. In washing the proteids care must be taken that they remain well spread over the surface of the filter, or in drying they may run into a mass from which it is almost impossible to eliminate both the fat and the moisture.

The greatest difficulty which presents itself in the analysis of condensed milk is estimating the cane and milk-sugar. If the gravimetric or volumetric methods be employed, the milk-sugar must be always estimated before inversion, and if mineral acid be used for inversion, allowance be made for the change in the copper reducing power of the milk-sugar that follows its inversion. The precautions to be observed in estimating by means of Fehling's solution have been fully described by Soxhlet.

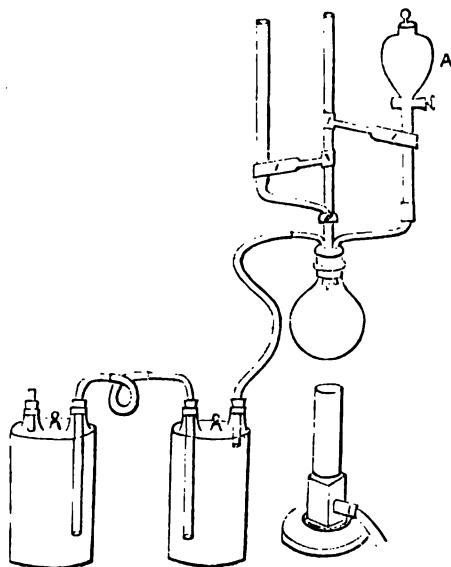
A method has been described by Messrs. Stokes and Bodmer by which they avoided the inversion of the milk-sugar, by employing a dilute solution of citric acid for the



inversion of the cane-sugar, and adopting Pavy solution for estimating both sugars, but their method for inverting the cane-sugar by citric acid can of course be equally well used in conjunction with Fehling's solution.

It appeared to me that the difficulties accompanying the inversion of sugars could be entirely evaded by a process combining the use of the polariscope with the volumetric or gravimetric method, and my method having thus far given satisfactory results I will describe it in detail.

Dilute 30 grammes of condensed milk, boil and when cool make up to 97 c.c. by the addition of water, then add 3 c.c. of acid solution of nitrate of mercury. If now the mixture be poured to and fro between two beakers the proteids will coagulate almost immediately, and very uniformly, yielding a perfectly bright whey on filtration. To



prevent a possibility of birotation it is essential to heat the solution to boiling point, for condensed milk will by microscopical examination be found to contain two forms of crystalline sugar: (a) cubical crystals, presumably cane-sugar, (b) crystalline needles, most likely milk-sugar.

Immediately sufficient whey has been separated 10 c.c. of it are diluted to 100 c.c., and retained for estimating the milk-sugar, whilst another portion should be at once examined by the polariscope, for after addition of the mercury solution, the inversion of the cane-sugar commences, and although during the few minutes necessary for completing these operations, the loss is inappreciable, if the process be delayed unduly, loss might occur. I have, however, satisfied myself that a diluted whey containing only 0.3 p.c. of the mercury solution may be kept for a short period without appreciable change; in practice, therefore, I conduct this operation last, though for convenience I describe it first.

The diluted whey may be titrated either with Fehling solution or with Pavy solution. I have found so far that the latter gives with milk-sugar very reliable results, and it offers some advantages over Fehling. One great disadvantage attached to the Pavy process has been that as soon as all the ammonia escapes from the solution oxidation commences, thus frequently bringing the experiment to an untimely end. I have surmounted this difficulty by a modification of the apparatus used by Messrs. Stokes and Bodmer, which I proceed to describe. I titrate the dilute whey with 40 c.c. of Pavy solution, the formula for which is given below, using the following modification of the apparatus usually employed. Fig. 1 shews that the modification consists of the reservoir B. containing strong solution of ammonia, and connected with the flask D. containing the Pavy solution; by means of this the ammonia driven off from the latter can be replaced, and the process continued for an indefinite period. It is necessary the burette be placed as shewn in the figure, that it may be well removed to one side of the flask to avoid an error due to the expansion of the contents of the burette.

The Pavy solution was standardized with milk-sugar, the purity of which was previously determined with the polariscope: so much of the whey is at first introduced from the burette into the flask containing the cupric solution as is expected should change the colour in that solution, without completely decolouring it. If there be no clue to the amount necessary for this purpose, the whey may be dropped in slowly until the change in colour is evident. Then before adding more whey one must boil until the colour remains constant, introducing fresh ammonia if necessary from time to time. The change in colour proceeds at first fairly quickly, but later very slowly. I find it is usually six or seven minutes before the colour can be said to be constant, and in order to be on the safe side I continue the boiling for about six minutes, then add more whey and again boil for six minutes, completing the process when the colour is reduced to an almost imperceptible blue, and after the boiling has been continued for *ten* minutes subsequent to the last addition of whey.

In the case of milk-sugar I find the results are not affected by the rate at which the whey is added.

It is not necessary to neutralize the diluted whey, for I have found that the presence of so small a quantity as 0.3 per cent. of the acid solution of nitrate of mercury does not appreciably affect the result. I have invariably obtained correct results by the first titrations, but confirm them by a second titration, adding the full amount at one time.

A portion of the whey is now examined by the polariscope; the indications on the scale of that instrument will be due to the combination of cane-sugar with milk-sugar, but a deduction can be made corresponding to the amount of the latter found by the Pavy test.

It is also necessary to correct both for the space occupied by the fat, and the proteids, using the following formulæ upon the data given by Dr. P. Vieth.

Fat: supposing our sample contains 10 per cent. fat by weight, the necessary calculations to obtain the percentage of fat by volume is as follows:—

$$93 : 100 = 10 : x$$

$$x = 10.75.$$

*It was pointed out by Dr. P. Vieth that milk contains 3.5 to 4 per cent. proteids,*



hence in estimating the sugar he allows 3 per cent. for the space occupied by these. I find that if I calculate that every 3.75 per cent. by weight of proteids in a condensed milk are the equivalent to 3 per cent. by volume after making the necessary calculations for these and for fat, the percentage of sugar found is approximately correct; therefore the following is the necessary calculation for the space occupied by the proteids, supposing that they are found to be 11.25 per cent. by weight:—

$$5:4=11.25:x \qquad x=9$$

I get more accurate results by estimating the space occupied by the proteids and the fat in this manner, than by washing the precipitate and adding the washings to the whey, both because the latter gives too dilute a solution to give satisfactory results, at any rate with my polariscope,\* and further because the results are apt to be vitiated by the sugars becoming more or less inverted, when left for any considerable time in contact with mineral acid.

In order to test the accuracy of my process, I prepared some solutions of cane-sugar in milk, and proceeded as in analysing a condensed milk. The following are the results:—

	Added.	Found.	Error.
Cane-sugar	8 grms.	7.94 grms.	— .06
	9.2 „	9.08 „	— .12
	10.5 „	10.59 „	+ .09
	9.5 „	9.6 „	+ .1

The following is a series of complete analyses of condensed milk:—

	1	2	3	4	5	6	7
Moisture ..	30.3	24.8	26.8	28.	26.4	25.6	26.8
Fat ..	4.7	4.7	4.7	4.	11.5	10.4	10.65
Proteids ..	12.6	12.4	12.37	12.1	12.6	10.67	11.07
Ash ..	2.1	2.43	2.27	2.37	2.05	1.9	1.9
Milk-sugar ..	15.28	15.72	14.6	14.9	14.4	14.52	14.2
Cane-sugar ..	35.15	38.7	39.9	38.7	29.95	33.59	32.15
Total ..	100.13	98.74	100.64	100.07	96.9	96.68	96.77
Error ..	+ 13	— 1.26	+ .64	+ .07	— 3.1	— 3.32	— 3.33

The first four samples are brands of condensed skim milk, and the results are very close, but there is a loss in the last three samples, which are brands of condensed milk from which no cream has been abstracted. I believe that this loss may in part be accounted for by the 70 or more per cent. of greasy solid matter retaining a portion of the moisture.

My process does not entirely surmount the inaccuracies which would be caused by the presence of invert sugar in condensed milk. I must confess to seriously questioning

\* The Mitscherlich half-shadow polariscope described by Dr. P. Vieh (ANALYST, vol. xi., p. 161).

whether invert sugar occurs in more than traces at all frequently. Though familiar with the process of milk condensing, I do not know of any cause that will account for the presence of invert sugar, but should it be present an approximate idea might be obtained as to the amount present.

The existence of 2 per cent. of invert sugar in the sample would cause the cane-sugar to be estimated at 0.78 per cent. too low, and the total constituents of the sample to be estimated at 97.22 instead of 100.

Invert sugar having about double the reducing action upon Pavy solution than that of milk-sugar, the presence of 2 per cent. invert sugar would raise the percentage of milk-sugar found by about 4 per cent., the total of the combined sugars found being about 2 per cent. too high, thus compensating for the loss to total solids on the scale of the polariscope. Now the normal proportion of milk-sugar to proteids in milk is not likely to exceed 5 : 3.3 or in condensed milk say 15 : 10. If therefore the milk-sugar was raised to so high a proportion as 19 : 10, one would immediately suspect the presence of not less than 2 per cent. of invert sugar.

#### FORMULÆ.

##### *Solution of sulphate of copper—*

Crystallised sulphate of copper	..	..	..	34.64 grms.
Distilled water to	..	..	..	500 c.c. dissolve.

(N.B.—This solution is of the same strength as that used in preparing Fehling's solution.)

##### *Solution of Nitrate of Mercury—*

Mercury	..	..	..	50 grms.
Nitric acid (sp. gr. 1.42)	..	..	..	100 "
Distilled water	..	..	..	a sufficiency.

Dissolve the mercury in the nitric acid, and dilute the resulting solution by an equal bulk of the water.

##### *Pavy Solution—*

Crystallised sulphate of copper	..	..	34.65 grms.
Rochelle salts	..	..	170 "
Caustic potash	..	..	170 "

make up to one litre with distilled water : 120 c.c. of this fluid with 400 c.c. of ammonia (sp. gr. 88) are made up to one litre.

#### REFERENCES.

Report upon Soxhlet's researches on the quantitative estimation of the sugars by C. H. Hutchinson. *Pharm. Journ.*, 1881, pp. 722 and 757.

Notes on the estimation of milk-sugar in milk by means of the polariscope by Dr. P. Vieth, F.C.S., F.I.C. *ANALYST*, vol. xiii., p. 63.

The determination of mixtures of milk-sugar and cane-sugar by W. W. Stokes, F.C.S., and R. Bodmer, F.C.S. *ANALYST*, vol. x., p. 62.



## DISCUSSION.

Dr. VEITH, in reference to the remark of the author that the whey which he produced by adding nitrate of mercury was boiled before being put into the polariscope in order to avoid birotation, said he had never noticed birotation taking place under such circumstances in ordinary milk, and he did not think it was so with condensed milk. He did not quite catch what Mr. Shenstone meant when he said the proportion of milk-sugar and proteids was as 5 to 3.3. 5 was rather high for milk-sugar, and 3.3 would be very low for proteids. In milk containing 5 per cent. of milk-sugar he should expect more than 3.3 of proteids—3.8 or 3.9 would more likely be present. The analyses given were very interesting. The most extraordinary point was, in the last three cases, the large error which was not accounted for. If anything were going on in the case of whole milk, why should it not happen with the skim milks as well? The only other point which perhaps wanted some explanation was the ash, which in some cases was found rather low. It was a notable fact that the ash in milk stood in a very close relation to the solids-not-fat. It generally amounted to 8 per cent. of the solids-not-fat, and he had found this proportion in almost every case. Thus, in the first case, instead of 2.1 it should be 2.4; in the three last cases it was also too low—2.05 should be 2.16, and 1.9 should be 2.0. It was only a small difference, but still, in four cases out of the seven the ash was too low. The whole method which Mr. Shenstone followed in the analysis seemed to him (Dr. Veith) rather cumbersome. He had never found any difficulty at all in analysing condensed milk by simply making a solution in such a proportion that he got the constituents about the same as in ordinary milk. For instance, he would take one part of the condensed milks in question and dilute with two parts of water, and treat this solution like ordinary milk—that was the simplest way of doing it.

Mr. BODMER asked how long was Mr. Shenstone able to continue the boiling by adding ammonia. In regard to the point mentioned by Dr. Veith, he quite agreed with Mr. Shenstone that it was necessary to make a thorough mixture of the condensed milk to begin with. Either clots formed as the author remarked, or else the casein was not equally distributed through the milk; but, anyhow, he found it necessary to thoroughly mix the condensed milk before taking a sample for analysis.

Mr. DAVIES said he had been favoured with a preliminary account of the mode adopted by Mr. Shenstone and had found it to answer admirably. He had tried it side by side with the Stokes and Bodmer process, and of course had required to repeat the process two or three times in order to get a reliable result, but having obtained that, it was, he found, always possible to obtain it at one operation by the modification suggested. It was, as Dr. Vieth said, a very extraordinary circumstance that with the whole milks the numbers should have come out so considerably below the 100 per cent. The explanation of Mr. Shenstone had not entirely satisfied him as to the reason, but he could not himself offer a more satisfactory one at present.

Dr. MUTER (Chairman), after complimenting Mr. Shenstone on his paper, desired to make only two remarks. In the first place he would like to ask the author, if before setting to work to devise a new method, he had ever tried the old process originated by him (Dr. Muter) and communicated to the Society in 1880 for the estimation of cane-sugar in milk. That process was then favourably received. Several prosecutions for the addition of cane-sugar to ordinary milk (to cover its dilution) had been successfully brought upon its results. It did not require the use of any polariscope or other expensive instrument, and the sugar itself was directly weighed. With certain modifications, suggested by experience, it was in daily use and answered all the purposes required. In the second place he would advise Mr. Shenstone that in estimating his casein he would probably find it much better not to dry it before extracting the fat, but

to follow his washing water through with strong spirit, then extract with ether and dry once and for all.

Mr. SHENSTONE in reply said that as regards the necessity for boiling the solution of condensed milk, he had already called attention to the presence of two forms of crystalline sugar in that article; the crystalline needles were most likely crystals of milk-sugar; if so, as a fresh solution of this crystalline milk-sugar would cause an error through birotation, he recommended the precaution of boiling the solution. Natural milk contained no crystals of sugar, therefore the boiling would be unnecessary in that case. He was aware that the proportion of 5 per cent. milk-sugar to 3.3 per cent. proteids was very high as regards milk-sugar and low as regards proteids, but such a milk might occur, and therefore, unless the milk-sugar was found in a still higher proportion there would be no evidence of the presence of invert sugar.

Referring to the question: "Why should the error be greater in the case of samples rich in fat than in samples of skim milk?" he was not prepared with any further explanation at present; he might say that all his experiments so far had tended in that direction.

With regard to the ash being unusually low as compared to the proteids, he could only say he had incinerated the sample at a dull red heat with an argand burner, possibly the proteids were not quite uniformly mixed in the sample, notwithstanding his having taken the precaution of passing it through a wire sieve.

It appeared to him that the Chairman's method for estimating the sugars possessed some undoubted advantages. In practice he thought his own method would be found the most expeditious. He hoped to make some comparative experiments, examining the same sample by both methods, the results of which he thought should be very interesting. It was strange that Dr. Muter's method should have been so entirely overlooked by recent literature.

In reply to a question, Mr. Shenstone said he had continued the boiling of the Pavy solution for as long as half an hour, but could not say how much longer he could have continued it without oxidation.

Dr. VIETH asked Dr. Muter to give the details of his process, which had escaped the notice of many of the later members of the Society, owing to the difficulty of getting the earlier back volumes of the ANALYST.

Dr. MUTER said that to go into all details at the late hour at which they had arrived would be tedious, but he would, when Mr. Shenstone's paper appeared, give an abstract of the process as it appeared in the ANALYST, vol. v., and mention any subsequent improvements. In compliance with this promise he has contributed the following abstract:—

#### THE ESTIMATION OF CANE-SUGAR IN MILK.

By JOHN MUTER.

(For further particulars see the ANALYST, vol. v., pp. 35-40.)

TEN grams. of the milk are evaporated to dryness upon 4 grams. of hydrated calcium sulphate with frequent stirring, so that nothing sticks to the basin. The dry residue is powdered, placed in a dried filter and extracted with ether in the "Soxhlet," and the fat weighed as usual. The residue is transferred to a beaker, together with the filter containing it and 20 c.c. of hot (not boiling) water are added, and the whole is well stirred; 30 c.c. of rectified spirit (60° O. P.) are then added, and the mixture is allowed to cool, stirring occasionally. When cool it is thrown on a filter, placed over a long graduated measure, and washed with proof spirit until the filtrate measures 120 c.c. (usually sufficient unless the amount of cane-sugar be very large, as in condensed milks). The filtrate is divided into two equal parts, and the one portion is evaporated on the water-bath in a weighed platinum dish, and then dried at 212° to constancy, weighed, slowly burned to a white ash at a dull red heat and again weighed (weight—tare of dish + ash)  $\times 20$  = per cent. of total



sugars. In the other portion the milk-sugar is estimated by Fehling *gravimetrically* by the author's method of directly weighing the reduced cuprous oxide, and the milk-sugar so found  $\times 20$  = per cent. This deducted from the total = cane-sugar. In all milks containing over 2 per cent. of cane-sugar the process is accurate, but when dealing with small adulterations the following allowances must be made:

Per cent. of cane-sugar found.				Deduction to be made.			
Under	0.5	...	...	...	...	0.5	...
Over	0.5 but under 1.0	...	...	...	...	0.2	...
"	1.0	"	1.5	...	...	0.1	...
"	2	"	...	...	...	none.	...

Pure milk thus treated shows a slight difference (about  $\frac{1}{2}$  as a maximum) between the total sugar and the estimated milk-sugar, and therefore a limit of  $\frac{1}{2}$  is fixed, below which the process is not to be used, and it is *only to be applied when the presence of sugar can be distinctly proved by taste* in the original sample. A proper training of the palate can detect the addition of any amount of sugar to milk over half per cent.

As to the gravimetric Fehling and direct weighing as  $\text{Cu}_2\text{O}$ , although it is apparently against all preconceived ideas, it has still stood the test of time in the author's hands. He reiterates his opinions (1) That the only reliable reduction of copper obtained from milk-sugar is when the "Fehling" is added all at once and in distinct excess. (2) That it is quite possible to dry and weigh  $\text{Cu}_2\text{O}$  with sufficient accuracy, if only the method be practised. (3) That by this process the  $\text{Cu}_2\text{O}$  found multiplied by the factor 6835, gives the actual amount of milk-sugar as it really exists in the milk with sufficient accuracy for all ordinary purposes. The author now makes some slight modifications on the original process. Thus instead of a weighed filter as originally used for collecting the  $\text{Cu}_2\text{O}$ , a pair of tared filters are employed so as to equalise any possible action of the "Fehling" on the paper. Again the final washing water is displaced by strong spirit, and then that is in turn displaced with a little petroleum spirit before putting in the bath. The use of the thick pad of dry white blotting paper below the filter paper in the bath is absolutely necessary, and a slight pressure of the filter and contents between thick blotting paper before putting in the bath is now resorted to. A good quick-running filter paper should be used, and the rapidity of manipulation of the whole process should be aimed at. The author has never himself tried how far Pavy's method would do for the estimation of the lactose, but for those who prefer volumetric work, it might possibly answer sufficiently well. For an ordinary analyst, only meeting occasionally with sugared milks, however, the gravimetric process as detailed in the original paper (with the above modifications) is far the best and most convenient.

(Conclusion of Society's Proceedings.)

## ON THE RELATIVE VALUE OF DIFFERENT PEPSIN TESTS.

By JAMES H. STEEBINGS, JR.

(Concluded from page 212.)

2. We are further instructed to raise the temperature of the mixture after six hours' digestion up to and above  $145^{\circ}\text{F.}$  to destroy the peptic principle, and then the bath, with contained bottles, is allowed to rest over night. It has been shown by numerous experiments that the peptic principle is not killed at this temperature ( $145^{\circ}\text{F.} = 62.2^{\circ}\text{C.}$ ), but that, on the contrary, digestion may and does continue up to  $80^{\circ}\text{C.}$ , though of course much more slowly than at a lower temperature. If, therefore, a bottle containing a quantity of undissolved albumen be subjected to a temperature of  $62.2^{\circ}\text{C.}$ , and that only for a comparatively short time (or during the time that the water bath is cooling), it is manifest that the peptic principle would not be injured, and that, consequently, digestion will progress all night. We should thus obtain a much too high and an erroneous result.

3. The next step in the process is to pipette off 10 c.c. from the settled contents of

the test bottle, and evaporate to constant weight. Although I have found that, as a rule, the undigested albumen remaining in the test bottle settles pretty well over night, yet there is always a little left suspended in the liquid, which, if pipetted off, as it must be, would add to the weight of the dry residue obtained from the 10 c.c. pipetted off, and hence would be a new source of error.

4. We are next directed to evaporate the 10 c.c. to dryness, and in doing this the residue chars or carbonises, owing to the free hydrochloric acid contained in the water, so that in reality we weigh a certain amount of carbonaceous matter, instead of peptone, etc. Does this represent the amount of coagulated albumen dissolved by the pepsin? It may, but I strongly doubt it.

5. Assuming that the dry residue had not charred, what would we then be weighing? We would be weighing a mixture consisting of undigested albumen, partially-digested albumen, and fully-digested albumen, or peptone, together with all the intermediary products which always are to be met with in peptic digestions. Does this represent the efficiency of the pepsin, or is it not more likely that the amount of true peptone formed is an indication of the strength of the pepsin? The latter, in my opinion, is the more plausible explanation, especially if it be remembered that only the peptone is assimilable in the human system, while the intermediary products are not. In short, if we let the weight of all the products formed in a peptic digestion represent the efficiency of a pepsin, we would be obtaining a result considerably above what it ought to be.

6. As far as the calculations involved in this test are concerned, I would say that they are very ingenious and eminently fitted to make a pepsin test as high as possible. I will not dispute the accuracy of the multipliers 7 and 8, but do not believe that their accuracy is infallible in every test.

As this test was particularly got up to determine the digestive power of concentrated pepsins, I therefore append tests made by myself upon two such pepsins.

*Pepsin E.*—One grain of this pepsin was found to digest 819.2 grains coagulated egg albumen in six hours.

*Pepsin F.*—One grain of this article was found to digest 784 grains of coagulated egg albumen in six hours.

I have found by personal experience that the accumulation of peptone during peptic digestions, hinders, and finally stops, the action of the pepsin upon the albuminoid matter entirely. If now the liquid be diluted, digestion will recommence again, and proceed until the pepsin has become inert. (I do not believe in the theory that one grain of pepsin can go on digesting to infinity.)

As Manwaring lays particular stress upon this question of dilution, I think his test is a decided improvement over the U. S. P. test.

The next good point in his test lies in the fact that he does not attempt to weigh the undigested albumen, as is done in the U. S. P. test, and thereby does away with a great source of error; but instead of this he figures the amount of albumen (?) digested upon a dry basis, and then tries to convert this dry basis by calculation into albumen on the wet basis. In doing this errors are apt to occur, as I have already pointed out, but *I do not think that they are errors of such magnitude as are apt to be obtained with the U. S. P. test.*



Finally, I wish to say a few words about a test which I consider to be the only approach to an accurate method of testing pepsin that I know of. I do not claim that this test is absolutely accurate either, as slight errors are apt to occur, which, however, do not materially injure the final result. I refer to the Kremel test, which was published some time since in the Druggists' Circular.

In devising this test Kremel has made a radical departure from the usual methods, and bases his test upon the fact that under the conditions in which artificial peptic digestions take place, pepsin alone has the property of converting albuminoid matter into peptone, and that, therefore, from an analytical as well as from a physiological standpoint, the only correct method is to take the quantity of peptone produced as a gauge of the action of the pepsin; or in other words, the test is made to resemble as nearly as possible the conditions existing in the natural process.

Without going into any further detail the test is made as follows:

One grm. of egg albumen (soluble) dried at  $40^{\circ}$  C. and pulverised, and 0.1 grm. of the pepsin to be tested, are placed into a 100 c.c. flask, and dissolved in 50 c.c. of 0.2 per cent. hydrochloric acid. The solution is heated to  $38-40^{\circ}$  C. for three hours, and then exactly neutralised with sodium carbonate; it is then heated on a water bath to  $90^{\circ}$  C., and cooled after coagulation has taken place. The flask is then filled to the mark with distilled water, and 50 c.c. are filtered off and evaporated to dryness in a platinum dish on a water bath.

The residue is dissolved in hot distilled water, filtered through a moist filter into a platinum dish, and the filter carefully washed. The solution is again evaporated to dryness and weighed. The peptone is then incinerated with ammonium carbonate, and the weight of the ash deducted leaves the weight of the pure peptone, or the representative of the digestive power of the pepsin.

The good qualities of the above test are the following:—

1. Simplicity.
2. No guesswork, troublesome calculations, or the use of questionable factors.
3. No weighing of albumen dissolved in hydrochloric acid, undigested albumen and intermediary products along with the peptone. This is all obviated by the use of soluble egg albumen, coagulation and filtration or removal of the undigested portion as detailed above.
4. The ease with which it is possible to duplicate and still obtain concordant results.

On the other hand, the objections to this process are the following:

1. The great difficulty of procuring absolutely pure soluble dried egg albumen. This source of error, however, in my opinion, is very slight, because in each test a large excess of albumen is always used, and consequently the pepsin always has enough albumen to act upon. Besides this, it must be remembered that only the peptone formed is weighed, and not the amount of undigested albumen, as is the case with the U. S. P. test.
2. It may be objected to this test that the results obtained are expressed by the weight of peptone formed and not by the weight of albumen dissolved, and consequently the figures, being based upon dry peptone, will be much lower than when the result is

expressed as so much moist or coagulated albumen. If this, however, be objected to, it is comparatively easy to obtain higher figures by a simple calculation. Assuming that the amount of dry peptone obtained is equivalent to so much dry albumen, then by multiplying the weight of the latter by 8 (Manwaring's multiplier) we would obtain the equivalent in coagulated or moist albumen. I do not think it necessary or advisable to follow this course, as it involves the use of a multiplier which, as already pointed out, is questionable.

3. It takes a little longer to make a test by this process, but if accuracy is thereby gained the process is to be preferred.

To further illustrate the test, I append the following results, obtained with commercial pepsins:

								Peptone formed from 0.1 gm. pepsin in 3 hours.
Pepsin	G	..	..	..	..	..	..	0.5844
"	E	..	..	..	..	..	..	0.4972
"	B	..	..	..	..	..	..	0.4722
"	F crystal	..	..	..	..	..	..	0.4682
"	C (saccharated)	..	..	..	..	..	..	0.4676
"	H	..	..	..	..	..	..	0.4598
"	A (saccharated?)	..	..	..	..	..	..	0.4370
"	A (saccharated)	..	..	..	..	..	..	0.4246
"	D plain, soluble	..	..	..	..	..	..	0.3470
"	D pure, scales	..	..	..	..	..	..	0.3250
"	D pure, another sample	..	..	..	..	..	..	0.3146
"	I (saccharated)	..	..	..	..	..	..	0.2780
"	J French	..	..	..	..	..	..	0.1848
"	K (saccharated)	..	..	..	..	..	..	0.1738

These tests were all made with the same quantity of pepsin, whether the latter was saccharated or not, and, I think, are a fair indication of the relative values of the different pepsins.

It may be objected that this test does not do a concentrated pepsin full justice, on the ground that the latter would form a much greater proportion of peptone and thus retard if not completely arrest any further action of the pepsin upon the albuminoid matter.

In order to test this question, I saccharated samples of E, F and H respectively, according to Manwaring's directions, which is equivalent to diluting with mere acidulated water, and submitted them to the same conditions as before, and obtained the following results:

								Peptone formed from 0.1 gm. pepsin in 3 hours:
Pepsin	E	..	..	..	..	..	..	0.2620
"	F	..	..	..	..	..	..	0.1240
"	H	..	..	..	..	..	..	0.1250

It will be observed that in these tests the figures are considerably lower than in the former ones; but it must be remembered that the pepsins with which the tests were made were twenty times weaker, or rather more diluted than in the previous tests, and notwithstanding this the peptone formed is proportionally larger than before. This



ould clearly show that dilution is beneficial in the case of concentrated pepsins, as it rectes the retarding action of peptone. As the dilution in these last tests was twenty es greater than in the previous ones, we ought, by multiplying each of the above ults by twenty, to obtain the amount of peptone which would be formed by using pepsins in their concentrated forms, viz. :

								Peptone that should be formed from 0.1 grm. concentrated pepsin in 3 hours.
Pepsin E	..	..	..	..	..	..	..	5.240
" F	..	..	..	..	..	..	..	2.480
" H	..	..	..	..	..	..	..	2.500

The above figures are not, however, obtained, as has already been shown, and there- e the calculation is erroneous.

As all the results obtained by strictly following Kremel's directions are com- ble among themselves, I do not see how the process can well be improved upon.

The mere fact that the increased dilution increases the yield of peptone is not, in opinion, sufficient reason for condemning the process. As the conditions prevail- g in the stomach of a full-grown man do not differ materially as to dilution from day day, it is safe to say that pepsins of varying strength administered to such a person ll only perform a certain amount of work and no more, and that, consequently, the ults obtained by this test more closely resemble the conditions prevailing inside the mach than any other.

In conclusion, it will be seen that all the tests mentioned in this paper are subject faults and imperfections, some having more than others ; and, therefore, all we can under the present unsatisfactory state of affairs is to select the one which is least jectionable, and this, in my opinion, is the Kremel test.

## N SOURCES OF ERROR IN DETERMINATION OF NITROGEN BY SODA- LIME, AND MEANS FOR AVOIDING THEM.

By W. O. ATWATER.

(Continued from page 215.)

*es of Nitrogen in Distillation Products as indicated by Colouration of Acid Solution in the Nitrogen Bulb.*

It is frequently urged, *e.g.* by Johnson and Jenkins,\* that the combustions should so conducted as to avoid any considerable discolouration of the acid solution, since the ouring matter may contain nitrogen. I have been at no little pains to learn how to ure this result. In how far it may be accomplished by the use of slaked lime, as ggested by Johnson, I am unable to say, having had no experience. One of my istants, Mr. G. P. Merrill, made, at my suggestion, a considerable number of combus- ns with nitrogen-free stearin at different temperatures, with varying degrees of idity and different lengths of anterior layers of anterior soda-lime. In general we ind that the more the gaseous products of distillation in the tube were brought into act with the soda-lime, the less was the discolouration manifested. But we were un-

\* Report of Conn. Ag'l Exp't Station, 1878, 116.

able, even with the highest heat, very long tubes, and slow conduct of the operation to secure a complete combustion of the distillation products; materials with the odorous hydrocarbons being always given off in considerable quantities. These and other experiences gave me the impression that the largest conveniently practicable distance between the gaseous products of distillation and the soda-lime was desirable, and on account we practised for a considerable time the use of long tubes with a long layer of soda-lime. Several circumstances, however, have shaken our faith in the need of this precaution. One is found in the comparative results of determinations with the long and the short tubes detailed in Article IV. of this series, in which the same quantities of nitrogen were obtained with tubes of 40 cm. and closely packed anterior layer of soda-lime of 12-15 cm., as with tubes of 60-75 cm. and correspondingly longer anterior layers. The comparative results in the combustion of ammonium sulphate in the long and short tubes, cited in the same article, seem to preclude the assumption that a loss of nitrogen from dissociation in the long tubes could have compensated for what otherwise have been a gain from more complete ammonification. This effort to secure more perfect ammonification by means which tend to prevent the formation of carbon compounds was therefore without avail. Another fact which militates against the necessity of that colouration of the solution necessarily indicates the presence of nitrogen compounds which have escaped ammonification, is found in the results of quite extensive experiments in this laboratory, in which we have taken pains to note through a period of several months the amount of discolouration in the acid solution as measured by the eye. When we came to collate these observations, which include many scores of determinations, we found to our surprise that we had, with no exceptions exceeding the practically usable variation of duplicate analyses, as much nitrogen with the considerably discoloured and turbid as with the nearly clear and colourless solutions; nor did the quantity of nitrogen average materially less in the former than the latter.

Johnson mentions that in combustions with the soda-lime made from slaked lime and sodium carbonate, as suggested by himself, "the acid in the bulk-tube is frequently coloured more or less deeply red." On one occasion, in making some combustions with Johnson's soda-lime, we noticed that the acid in the bulb became reddish in colour during the combustion. The soda-lime was a little moist. It was dried and the same combustions repeated. The solution then remained perfectly colourless. On another occasion I noticed a similar red colour in the solution when moist ordinary soda-lime was used. The same soda-lime was dried and the combustions repeated. The solutions were then colourless. In each of these two cases the solution was very clear with both the moist and the dry soda-lime, and the red colour with the moist soda-lime was very pronounced. What the colouring matter was I am unable to state, but the observation seems very similar to the one by E. Salkowski, above cited, from which he infers the presence of the chromogen of urobilin.

The following series of determinations of nitrogen in a specimen of (probably impure) albumen prepared from beef, include the observations just referred to, and with Johnson's soda-lime, and may be worth citing.

The combustions were conducted at what we have called "high heat," to *very highest heat* the tubes of the most difficultly fusible Bohemian glass we could



would bear without bursting. The time of the combustions was about 60 minutes in each case. The percentages of nitrogen are calculated on water-free substance. The determinations were made by a student in this laboratory some years ago, when the investigations here reported were being entered upon. The results are given in Table I.

The differences in results, which vary from 15.71 to 16.24 per cent., I presume to be due to differences in the charging of the tube. That is to say, it seems to me most probable that the low results in Nos. 3-8 were due to loose packing of the soda-lime in the tube, and consequent incomplete contact of distillation products with the soda-lime. I can think of no other explanation of the wide discrepancies. There is perhaps a relation between either the clearness or the colour of the solution and the apparent accuracy of the results, though it is not very pronounced.

TABLE I.  
*Determination of Nitrogen in Albumen.*

Number .. ..	1	2	3	4	5	6	7	8
Soda-lime .. ..	Ordinary.		Johnson's.			Ordinary.		
Length of tube .. ..	75 cm.	75 cm.	75 cm.	75 cm.	40 cm.	40 cm.	40 cm.	40 cm.
Solution .. ..	Nearly clear. Colourless.		Perfectly clear. Deep red.		Somewhat turbid. Colourless.			
Per cent. N found ..	16.24	16.25	16.05*	16.07*	16.00	15.78	15.71	16.10

\* Soda-lime moist.

Thus in Nos. 1 and 2 the determinations were apparently correct (though there is no proof that such was the case). The solution was colourless and had but little matter in suspension. In Nos. 3 and 4, in which the soda-lime was moist, there was no visible suspended material, but the solution was red, and less nitrogen by about 0.2 per cent. was obtained. In Nos. 5 and 6, which were duplicates of Nos. 3 and 4, except that the soda-lime was dried, the solution was colourless but somewhat turbid from suspended matters, and still less nitrogen was obtained. In Nos. 7 and 8, which were duplicates of Nos. 1 and 2, except that the tubes of the latter were of extra length, the solution was somewhat turbid and the percentages of nitrogen were small.

When these results were obtained we were inclined to interpret them as confirming the idea that turbidity and colour in the solution implied loss of nitrogen. But perhaps all that they amount to is to furnish illustrations of the general facts that neither agreement of duplicates (compare 1 and 2 with 3 and 4) nor the clearness or turbidity, nor the presence or absence of colour of the solutions, can be taken as a test of the correctness or incorrectness of the determinations.

Although we were somewhat surprised to find, in comparing the large number of results referred to above, that the nitrogen obtained where the solutions were nearly clear and colourless averaged just about the same as where they were more turbid or coloured, yet it is after all not so strange, in view of the facts that :

(1) Non-nitrogenous materials, such as the decomposition products of fats and carbohydrates, and probably the non-nitrogenous cleavage products of protein compounds as well, may impart turbidity and colour to the solution. That is to say, neither colour nor turbidity is at all an evidence of presence of nitrogenous matters.

(2) Even if the colour be due to nitrogen compounds, it does not by any means prove the presence of enough to have any material effect upon the result, since the quantity which would colour a small amount of solution might easily be far too slight to be revealed by titration.

(3) Many volatile nitrogenous compounds such as we should expect to escape ammonification in soda-lime combustions, *e.g.* amido acids and amines, are colourless and soluble. Hence absence of turbidity or colour is no proof of absence of non-ammonified nitrogen compounds.

I do not by any means mean that turbidity and colour are not to be avoided if practicable, or that they do not indicate imperfect combustion. What I wish to urge is, that neither colour nor turbidity, unless excessive, is an indication of a bad, or their absence a proof of a good determination.

The danger of loss of nitrogen through oxidation by nitrates occurring in the soda-lime as impurities, or introduced with the substance to be analysed, requires no discussion here.

What has been urged regarding incomplete ammonification of nitrogen of protein compounds may be briefly recapitulated as follows :

(1) Loss by incomplete decomposition would seem to be best prevented by fine pulverisation of substance, thorough mixing with plenty of soda-lime, and heating until no considerable amount of charred residue is left.

(2) Loss by formation of cyanides which combine with the bases of the soda-lime, does not seem to occur if the substance is mixed with enough soda-lime of proper water content and properly heated. Comparison of results of properly conducted combustions with those obtained by other methods implies no considerable escape of nitrogen in the free state.

(3) There is great danger of loss of nitrogen in volatile distillation products which escape ammonification. With some compounds, as alkaloids and leucine, this seems extremely difficult to avoid; but with the ordinary protein compounds of animal and vegetable tissues, and with the casein of milk, the experience of this laboratory implies that complete ammonification can be insured by providing for sufficient contact of the substance and its decomposition products with heated soda-lime, (*i.e.* with water vapour at high temperature).

(4) This needed contact is best secured by (a) thorough mixing of substance with soda-lime; (b) use of soda-lime which contains a rather large proportion of lime, is not too fusible and does not shrink too much in heating; (c) carefully avoiding a channel; (d) providing a considerable anterior layer of soda-lime; (e) heating this latter to dull redness before bringing the heat to bear upon the substance, and keeping it hot until the combustion is done.

(5) While it is desirable to avoid the escape of matters which colour the acid solu-



in the nitrogen bulb and render it turbid, the presence of these is not an indication incomplete, nor their absence an indication of complete ammonification of the oxygen.

(*To be continued.*)

## NOTES UPON METHODS FOR ESTIMATING THE QUANTITIES OF HOMOLOGOUS ACIDS PRESENT IN ARTIFICIAL SALICYLIC ACID.

BY ERWIN E. EWELL AND ALBERT B. PRESCOTT.

(*Concluded from page 210.*)

### II.—A Method by Conversion to Phenols.

When salicylic acid and its homologues are distilled from lime they yield a distillate of their respective phenols, the elements of carbon dioxide being retained by the lime, so far becoming a carbonate. Subjecting commercial salicylic acid to this reaction was undertaken to apply to the distillate a limit test for composition of the produced phenols—namely, the test\* by adding an equal volume of 9 per cent. solution of sodium carbonate, and then noting the number of volumes of water to be added to cause beginning precipitation. Experiments were undertaken, with mixtures of a fairly representative salicylic acid and good carbolic acid, to obtain limits of dilution for each 5 per cent. dilution of cresol in the mixture.

A cresylic acid of the market, of specific gravity 1.04, was found to yield results so consistent that it was taken as an approximately representative cresol. Best carbolic acid of the market, with water just enough to liquefy it, was taken as phenol. From mixtures of these imperfectly pure articles preliminary data were obtained, as set forth in the following table:—

Volume per cent. of cresol in the distillate.	Calculated weight per cent. of hydroxy-toluic acid distilled.	After adding an equal volume of 9 per cent. sol. of soda, number volumes of water added before precipitation.
5	4.9	6.7
10	9.8	6.0
15	14.8	5.25
20	19.8	4.5
25	24.7	4.0
30	29.7	3.6
35	34.7	3.3
40	39.7	3.1
45	44.7	2.8
50	49.7	2.6

The conversion of the salicylic acid into its corresponding phenols was done as follows: 15 grms. of the acid and an equal weight of lime are thoroughly dried, well triturated together, placed in a glass retort, put over a strong heat, and quickly distilled,

\* A. H. Allen, 1878: *The Analyst*, 3, 321; Allen's "Commercial Organic Analysis," 2, 551; Lunge's *Coal Tar Distillation*, 63

collecting the distillate in a well-cooled receiver. To promote the distillation with great advantage, dried iron filings may be added in equal quantity to the contents of the retort. When the distillate is complete, it is liquefied by adding just enough water.

The sample of commercial salicylic acid previously tested by the method of acidimetry was subjected to this process, and the distillate tested by an equal volume of 9 per cent. sodium hydrate, and subsequent dilution with measured water, until after stirring there remained visible precipitation. Five volumes of water were required, indicating, according to the table above, some proportion of hydroxy-toluic acid between 14.8 and 19.8 per cent., and agreeing substantially with the result by acidimetry.

It appears evident that a method by conversion of phenols can be made effectual for the estimation of homologous acids in the salicylic acid in use, and probably with closer results than those obtained by acidimetry.

### III.—*Separation by Solubility of the Calcium Salts. The Method of Williams.*

This method, cited in the beginning of these notes, was the basis of the only estimation of the quantity of homologous acids in artificial salicylic acids which has come to the notice of the writers, and they have submitted it to a careful trial. The operation directed by Williams\* was conducted three times successively. The products of each operation—that is, the salicylic acid of assumed purity on the one hand and the homologous acids assumed to be free from salicylic acid on the other hand—were subjected to estimation by the method of acidimetry.

In each of the three operations the salicylic acid was obtained from the crystals of calcium salicylate in excellent crystals, and in each case acidimetry gave results for pure salicylic acid. The mother liquors, treated for separation of the homologous acids,† yielded acid agreeing with that described by Williams, acid differing greatly from salicylic in physical properties. This “homologous acid,” subjected to acidimetry, gave, for 1 grm. of the acid, (1) 705 c.cm., (2) 699 c.cm., and (3) 697 c.cm. of the hundredth-normal solution of alkali, the average being 700.3 c.cm. By the average of these estimations, then, the “homologous acid” still retained 61 per cent. of salicylic acid, against 39 per cent. of hydroxy-toluic acids, and in estimation (3), where the separation was carried further than in the other two trials, there remained over 50 per cent. of salicylic acid. Indeed, the process of crystallisation does not promise well for estimation, unless by some system of allowances and for an approach toward exact determination.

\* 1878: *Phar. Jour. Trans.* [3], 8, 785; *Proc. Am. Phar. Asso.*, 26, 536.

† The acid not salicylic has usually been designated in the singular number, and it may be that only one homologous acid occurs in the manufactured article. But as there are three isomeric hydroxy-toluic acids, to say nothing of the hydroxy-xyleneic, and in absence of any identification of these isomers in “homologous acid,” it is well to recognise their existence in the plural. Melting points of the hydroxy-toluic acids have been reported as follows:—

Acid—CO <sub>2</sub> H : OH : CH <sub>3</sub> = 1 : 2 : 3—	melting at 160° C.
Acid    "       "       " = 1 : 2 : 4—	"       " 173° C.
Acid    "       "       " = 1 : 2 : 5—	"       " 151° C.



## MONTHLY RECORD OF GENERAL RESEARCHES INTO ANALYTICAL CHEMISTRY.

THE DETERMINATION OF BENZENE IN A MIXTURE OF GASES. F. P. TREADWELL AND H. N. STOKES. *Berichte*, Nov. 12, p. 3131.—Using Berthelot's method (*Compt. Rend.*, 83, p. 1255), i.e., absorbing  $\text{CO}_2$  with caustic potash, unsaturated hydro-carbons with bromine water, and benzene with fuming nitric acid, the authors obtained discordant results. They find that the nitric acid used oxidises some of the CO present to  $\text{CO}_2$ , which it absorbs (see Hasenbach, *Journ. pr. Chem.* II., 4, 1), and also that the bromine water dissolves some benzene. Drehschmidt (see *Jul. Post's Chem. Techn. Analysen*, p. 108, 1888) previously obtained similar results. A. L. G.

THE ELECTROLYTIC PRECIPITATION OF COPPER. FR. RUDORFF. *Berichte*, Nov. 12, 1888, p. 3050.—As is well known, copper is best precipitated electrolytically in nitric acid solution. When chlorides are present the addition of nitric acid is disadvantageous. The author, therefore, attempted to electrolyse ammoniacal solutions, but obtained spongy precipitates. He obtained a hard, bright precipitate by adding 2 to 3 grammes of potassium or sodium nitrate to the solution, and then about 10 c.cm. of ammonia to each 100 c.cm. of solution containing 0.1 to 0.3 gramme copper. A. L. G.

PRESERVING SOLUTIONS OF TARTARIC OR CITRIC ACID. C. REINHARD. *Zeitschr. f. angew. Chemie*, No. 22.—Watery solutions of these acids, as is well known, do not keep well, and therefore it is the custom in most laboratories to keep the acids in the solid state and not to dissolve them until wanted. This inconvenience may be avoided by the addition of a small quantity of salicylic acid. The solution of tartaric acid employed by the author is made by dissolving 100 grammes of the crystals in one litre of water and .1 gramme of salicylic acid. For citric acid solution he uses twice as much salicylic acid. These small quantities of salicylic acid, which cannot possibly interfere with any analysis, are yet sufficient to keep these solutions for years. It is, however, astonishing that salicylic acid does not preserve a solution of sodium succinate. L. DE K.

ANALYSIS OF MEAT REFUSE MANURE. J. KONIG. *Zeitschr. f. angew. Chemie*, No. 22.—Owing to the great difficulty of getting a proper sample it has often happened that there was a serious difference in the percentage of nitrogen got by different operators. This may be to a great extent avoided by adopting the author's plan. 15 grammes of the manure (mixed as thoroughly as possible) are heated in a porcelain dish on the water-bath, with 150 c.c. of a mixture of 3 volumes of common sulphuric acid and two volumes of fuming acid. When everything has dissolved, the mixture is brought into a 200 c.c. flask, the dish is rinsed with acid and the whole finally made up to the mark. After proper mixing, 20 c.c. (= 1.5 grammes of manure) are now treated à la Kjeldahl, as usual. The advantage of this mode of proceeding, instead of weighing out about one gramme of the manure, is very plain. L. DE K.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

**STAINING COFFEE BERRIES.** J. KONIG. *Zeitschr. f. angew. Chemie*, No. 22.—Berries are often roasted with addition of roasting syrup, which unsavoury article, the author found to consist of: water, 26.21; fermentable sugar (dextro-glucose) 45.80; unfermentable bodies and dextrin, 37.45; ash, .54. Its use is said to be in preserving for a long time the delicate aroma of the berries, but, according to the author, it really constitutes a fraud; not only does it make the coffee decoction look stronger, but it enables the roasted berries to hold about 7 per cent. more water than they otherwise would

L. DE K.

**ARTIFICIAL COFFEE BERRIES.** J. KONIG. *Zeitschr. f. angew. Chemie*, No. 22.—A sample submitted to the author for analysis looked very much like the real article, only the berries had all precisely the same shape, which is not the case with the genuine product. The result of the analysis was as follows: Moisture, 5.14; nitrogenous matter, 10.75; fat, 2.19; non-nitrogenous matter, 76.76; woody fibre, 3.96; matter soluble in water, 29.88; ash, 1.20. Microscope revealed wheat starch. The berries, therefore, consisted no doubt of roasted wheat-flour dough of low quality. They are eagerly brought by retailers, and afterwards mixed to an extent of sometimes 50 per cent. with genuine berries.

L. DE K.

**BORACIC ACID IN WINE.** M. RIPPER. *Zeitschr. f. angew. Chemie*, No. 21, *Weinbau*, pg. 331.—The author found small quantities of boracic acid in a great many pure wines, also in the wood of the vine, so that it may be considered as a natural constituent of wine ash. It may be tested for as follows: 50 c.c. of wine are evaporated and burnt to ash in a platinum dish, and the ash treated with 10 c.c. of water. After adding 2 c.c. of hydrochloric acid a piece of turmeric paper is immersed and then dried on a watch-glass at 100°C. The characteristic brown colour will develop after 4 or 5 minutes. To make still more sure, expose the paper to ammonia gas or moisten it with solution of carbonate of soda, when it will turn a blueish-green black.

L. DE K.

## REWARDS FOR MERITORIOUS DISCOVERIES AND INVENTIONS.

We have been requested by the Committee on Science and the Arts of the Franklin Institute, of the State of Pennsylvania, to make known to our readers the fact that the Committee is empowered to award, or to recommend the award of, the following medals for meritorious discoveries and inventions, which tend to the progress of the arts and manufactures. The Elliott Cresson Medal (Gold) was founded by the legacy of Elliott Cresson, of Philadelphia, and conveyed to trustees of the Franklin Institute. By the Act of the Institution, May 17th, 1849, the Committee on Science and the Arts was designated and empowered to award this medal, and the Committee decided to grant it, after proper investigation and report by sub-committee, either for some discovery in the arts and sciences, or for the invention or improvement of some useful machine, or for some new process, or combination of materials in manufactures, or for ingenuity, skill, or perfection in workmanship.

The John Scott Legacy Premium and Medal (Twenty dollars and a medal of copper) was founded in 1816, by John Scott, a merchant of Edinburgh, Scotland, who bequeathed to the City of Philadelphia a considerable sum of money, the interest of which should be devoted to rewarding ingenious men and women who make useful inventions. The premium is not to exceed twenty dollars, and the medal is to be of copper, and inscribed "*To the most deserving.*" Full particulars will be sent on application to Mr. William H. Wahl, Secretary, Franklin Institute, Philadelphia.

## APPOINTMENT.

Mr. R. Bodmer, F.I.C., F.C.S., has been appointed Public Analyst for Bermondsey.























